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THERMAL DEGRADATION OF GRAPHITE/EPOXY COMPOSITES

H. L. PRITT

HERCULES INCORPORATED
AEROSPACE DIVISION
BACCHUS WORKS • MAGNA, UTAH

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The purpose of this effort was to develop visual, NDI, and residual strength correlations as a result of thermal degradation upon thick composite laminates. These investigations were performed upon dry 0.25-inch-thick, coated graphite composite (AS-1/3501-6) laminates subjected to thermal shock on one side. Exposure temperatures were 350°, 400°, 500°, 600°, 800°, 1200°, 1600°, and 1800°F. Temperature gradients were obtained from instrumental panels under no-load conditions. Thermal shocking of panels and specimens (see attached sheet)

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SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered) while under sustained strain (2500 μ in./in.) conditions provided comparisons of load carrying ability. Other specimens were tested for residual strength after thermal exposure. Ultrasonic examinations were performed, as was monitoring of color changes. Permanent matrix damage began to occur during a 600°F exposure.

FOREWORD

This document presents the final report for the effort performed by Hercules Incorporated on Thermal Degradation of Graphite/Epoxy Composites.

The Naval Air Development Center, Warminister, PA sponsored this study (Contract No. N62669-79-C-0270). Mr. R. E. Trabocca was project monitor.

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SECTION I

INTRODUCTION

The objective of this effort was to perform thermal degradation studies upon thick, coated graphite/epoxy laminates. The laminates were subjected to thermal shock at elevated temperatures. Visual and NDI examinations, as well as load testing during thermal shock and specimen testing after thermal shock, were to be used to establish an analytical correlation of the thermal degradation sustained at the various temperatures.

These thermal degradation studies were performed upon coated, 48-ply laminates of AS-1/3501-6 graphite/epoxy with an orientation of $[\pm 45/0_2/\pm 45/0_2/\pm 45/0/90]_{2s}$.

A data base was determined for the above laminate by testing unidirectional specimens at room temperature and 220°F.

All panels were examined visually and with ultrasonic "C" scan techniques prior to and after thermal exposure.

This investigation subjected the graphite composite test panels and specimens to thermal shock temperatures of 350°, 400°, 450°, 500°, 600°, 800°, 1200°, 1600°, and 1800°F. The various tests consisted of:

(1) Exposing laminates on the coated side with thermocouples embedded at various depths

(2) Exposing panels while under sustained compression strain

(3) Exposing specimens while under sustained four-point bend strain

(4) Rail shear testing of specimens after thermal exposure

(5) Compression testing of specimens after thermal exposure

(6) Reflective light measurements of exposed specimens

Data from the above tests were compiled to provide a correlation between damage and exposure temperature.

Sufficient 48-ply panels, 9 x 9-1/2 inch, (Figure 1) were fabricated with embedded chromel-alumel (Type K) thermocouples. Sensors were 12 plies, 24 plies, 36 plies, and 48 plies deep from the face which would be exposed to the elevated temperatures. Digestive analysis of specimens from this initial set of panels showed that fiber volume content was 61%.

One large, 48-ply panel was then fabricated and the various panels and specimens cut from it for the balance of the tests. This panel contained an average of 64.4% fiber volume.

All of these cured panels were lightly abraded, cleaned, and spray coated with a 0.0007- to 0.0009-inch-thick coat of MIL-P-23377, Type II epoxy primer on both sides. Two coats of MIL-C-81773, Type I polyurethane, Light Gull Grey (No. 36440), were then applied for a total of 0.0017-inch top coat thickness on the side of the laminate which would be exposed to thermal shock. All panels and specimens were dried at 225°F and ambient pressure prior to testing.

SECTION II

TESTING

A. COUPON TEST DATA

A summary of the AS-1/3501-6 coupon test data obtained at 220°F is presented in Table 1. All tests were performed on dry specimens in conditioning boxes mounted on Instron testing machines. Depending upon the property to be tested, specimens were either unidirectional, 90° transverse, or ±45° (in-plane shear properties). Where applicable, test data were normalized to 62% fiber volume. These coupons were made from three different prepreg runs with a different fiber lot in each.

B. ULTRASONIC INSPECTIONS

Ultrasonic "C" scan examinations were performed on all panels and specimens prior to thermal shock. These scans showed all panels to be of sound quality prior to thermal shock. Panels which had been subjected to thermal shocks when exposed to temperatures above 600°F were found to have major delaminations when examined ultrasonically.

C. INSTRUMENTED PANEL TESTS

A Lindberg Furnace, Model 51828, with a 12- x 12-inch door opening and a temperature capability up to 2012°F was used as the heat source for all testing at elevated temperatures. Incorporation of stainless steel sliding doors and ceramic insulation permitted thermal shocking the panels on one side.

During thermal shock tests, all panels were positioned so the panel side coated with the MIL-C-81773 material was exposed to the heat source. Temperature gradients during thermal shock exposures of horizontally mounted 9 x 9-1/2 inch, 48-ply dry panels are shown plotted in

Figures 2 through 10. Thermocouples positioned at three locations across the exposed face (1/16 to 1/8 inch off the surface) and embedded at depths of 12 plies, 24 plies, 36 plies, and 48 plies were used to obtain these temperature measurements. Temperature increments are listed in Table 2.

These tests were performed by stabilizing the oven temperature slightly higher than the desired exposure temperature with the sliding steel door closed. The instrumented test panel was then positioned over the sliding door, the door pulled aside, and ceramic insulation was packed around the panel edges to prevent heat loss. The backside of the panel was exposed to ambient conditions.

During panel exposure, a very faint (phenolic type) odor was first noticed during the 500°F exposure. During the 600°F exposure the odor was stronger. At exposures of 800°F and above, smoke was given off and the odors were very pungent. During a 1000°F exposure, an intermittent flame (approximately 6 inches high) was observed along one edge after 4.4 and 9.1 minutes exposure. This was not observed during a 1400°F and the 1800°F exposure. However, a 3-foot-high fireball occurred when the hot panel was lifted from the oven opening after the 1800°F exposure. This panel continued to burn around the edges (8- to 12-inch-high flame) for about 6 minutes in an ambient atmosphere.

Examination of air samples taken during the 1800°F exposure disclosed small charred particles (possible primer materials). No graphite fiber filaments were found floating in the air currents above the test panel.

The weight loss data generally follows what would be expected from visual observation of the panels. As the surface coating chars and fraying begins, the weight loss increases. (See Table 3.) Of interest is one 600°F panel which showed a major delamination by ultrasonics and only a small weight loss. Upon sectioning, the depth of the delamination was found to be 5 plies in from the coated face. The delamination, which was not readily apparent through visual inspection, could have a major effect upon structural integrity. When this panel was removed hot from the oven opening and laid aside in ambient air to cool, no visual evidence of structural degradation was present until after several hours at ambient conditions. Then, a raised area on the coated faces was observed. There is the possibility that the cool air shock (40°F) may have caused this delamination. Two other panels subjected to the same shock and handling did not show any signs of structural damage.

Table 4 shows the time at temperature for various exposures.

Ultrasonic and coating color changes due to thermal shocking are tabulated in Table 5. All panels exposed at temperatures above 600°F delaminated. Coating color changes began at the 400°F exposure.

Temperature measurements were hampered in some cases due to loss of thermocouples as a result of damaged leads. In addition, when the heating elements of the oven were on, the thermocouple which indicated oven temperature was noisy and provided data only when power was not flowing into the oven. To compensate for the 2-1/2-inch distance from oven thermocouple to panel surface, the oven temperature was purposely higher than the desired exposure temperature prior to opening of the sliding door.

Data for exposures from 350° to 1000°F were read manually and converted to millivolts and then to temperatures. Data for exposures above 1000°F were recorded on FM tape and a printout was converted to millivolts and then to temperatures. During the 1400°F exposure, the equipment recording internal panel temperatures was improperly set up, and data were obtained only briefly at the 5 and 10 minute intervals. This was corrected on the 1800°F exposure.

During exposure of 800°F and up, it appears that degradation effects (outgasing, delaminations, ablation) may create a cooling effect. For example, for the 1800°F exposure the oven temperature was 1880°F, but recorded temperatures near the exposed surface were much lower. (See Figure 10.) The exposure setup had no provision for sweeping degradation gases away from the surface of the panel as a result of the resin cooking off or charring.

Figure 11 shows thermal shock effects on some of these panels. No attempt was made to identify the time at which resin charring or delaminations occurred during the above exposures.

D. SUSTAINED COMPRESSION LOAD TESTS

Sustained compression load tests during thermal shock on one side of the panel at the various temperatures were performed in a Baldwin load test machine with the test panels mounted in a fixture as shown in Figure 12. The 4-1/2-inch-high by 9-inch-wide, 48-ply panel was mounted in the fixture with the 0° fiber orientation in line with the compression load. (See Figure 13.) Instrumentation locations are shown in

Figures 14 and 15. All panels were carefully aligned and lightly clamped along the top and bottom edges prior to application of loads and thermal shock. (See Figure 16.)

One instrumented panel was installed in the test setup under no compression load and subjected to temperatures of 350° to 600°F. This was done to verify instrumentation, recording equipment functions, adherence of the strain gages, and temperature effects on the strain gages on both sides of the panel. After the panel was subjected to 600°F for 16 minutes, the compression load was increased. Failure occurred at 37,900 pounds, with strain readings of 2628 μ in./in. on the cool side and 1192 μ in./in. on the hot side. Apparently, prior to load application, the panel had bowed slightly due to thermal gradients.

The planned test procedure for thermally shocking these panels from one side while they were under sustained compressive strain of 2500 μ in./in. was as follows:

(1) The panel was installed and lightly clamped in the fixture.

(2) Instrumentation was connected and verified as functioning.

(3) Recording equipment was checked out.

(4) Oven temperature was brought up to the desired level with door closed.

(5) Sustained load (strain) was applied and maintained.

(6) The sliding door was opened.

(7) Data were recorded while strain was maintained.

Those panels thermally shocked at temperatures of 350°, 400°, 450°, 500°, and 550°F while under 2500 μin./in. compressive strain withstood the test successfully. Posttest ultrasonic examination showed no evidence of thermal degradation. However, there was some discoloration of the coating as a result of exposures to 450°, 500°, and 550°F.

Those panels thermally shocked at 600°F and higher failed under this sustained compressive strain. (See Table 6.)

During thermal shock at 800°F, it was impossible to maintain strain at 2500 μin./in. It is suspected that the panel delaminated early in the test and deformed. The strain trace shows strain going up when the heat was reduced and load removed.

During the 1200°F thermal shock test, it was again impossible to maintain 2500 μin./in strain. Load and heat adjustments did not permit control of measured strain.

Maintaining strain at the 1600° and 1800°F thermal shock exposure did not present a problem due to the short exposure times. Test results may be seen in Figure 17.

Figure 18 shows the appearance of the panels after being subjected to these tests. Again, exposures above 600°F caused major disintegration on the panel face receiving the thermal shock. Exposures at 1200°, 1600°, and 1800°F showed that the laminate could carry the sustained strain for 2 to 3 minutes.

E. SUSTAINED FOUR-POINT BEND TESTS

Sustained four-point bend specimens were subjected to thermal shock in the Baldwin load test machine. Details of the setup and fixture arrangement are shown in Figures 19 and 20. These 1-1/2-inch-wide x 12-inch-long x 48-ply specimens were mounted in the fixture horizontally, coated side down, with the 0° fiber orientation parallel to the 12-inch length. A fixture with a length/thickness support ratio of 32 was built and used for this setup. Instrumentation details may be seen in Figures 21 and 22.

The procedure for performing thermal shock tests on the specimens while they were subjected to 2500 μ in./in. strain in a four-pount bending mode was as follows:

(1) The specimen was installed and aligned in the flex fixture.

(2) Instrumentation was connected and checked out.

(3) Recording equipment operations were verified.

(4) The oven was brought up to desired temperatures with door closed.

(5) Sustained load (2500 μ in./in. strain) was applied.

(6) The sliding door was opened.

(7) Data were recorded while strain was maintained or until failure.

Results are shown in Table 7 and Figure 17. Note that the loads imposed upon the specimens were increased substantially during the course of each test to maintain the desired 2500 μin./in. strain loading. Specimen failures occurred during thermal shock tests at 450°F and above. (During sustained compression load testing with thermal shock, failures occurred at 600°F and up.) Both the 1600° and 1800°F specimens deformed excessively due to resin matrix softening rather than fracturing.

After specimen 4P-1 had withstood the 400°F exposure with 2500 μ in./in. strain, the temperature was increased to the 450° range. The specimen withstood the higher exposure for 9 minutes under 2500μ in./in. strain before failing under a 1130 pound load.

Panel 4P-6 was then subjected to the 450°F shock, and structural load integrity was maintained for 16 minutes before failure occurred.

Based on similar failure load values, specimens shocked at temperatures between 450° and 1200°F appear to have suffered the same amount of thermal damage when failure occurred. Exposure times were shorter than those demonstrated in the sustained compression load testing. This may be attributed to higher loads imposed to maintain the sustained strain, the presence of shear stresses, and the fact that the neutral axis was shifting as a result of the temperature gradient through the specimen. In brief, it was a more severe test.

Three additional noninstrumented specimens which had not been subjected to thermal shock were taken to failure at room temperature for baseline information. Their average failure load of 1970 pounds may be used to compare performance of thermally shocked specimens. The specimens are shown in Figures 23 and 24.

F. RAIL SHEAR SPECIMEN TESTS

Panels were thermally shocked individually on the coated side at exposure temperatures of 350°, 400°, 450°, 500°, and 600°F. Exposures were maintained until backside (cool) temperatures stabilized. Ultrasonic examinations were performed and only one panel (T11 which had been shocked at 600°F) showed partial delamination. These panels were then cut in a specimen configuration as seen in Figure 25.

Originally, the gage size was determined by three main considerations. The first was the 0.25-inch width needed to install the shear strain gage. Second, analysis disclosed that the ratio of the gage length to width should be at least 12/1 to preclude combined loads from being imposed into the ungripped failure area. And third, the Instron test machine with conditioning box installed for testing at 220°F was limited to a maximum 20,000 pound pull. (During testing, this machine capacity was found to be only 16,600 pounds.) Thus, the minimum specimen size believed appropriate for these tests was as seen in Figure 26 using the test fixture shown in Figure 26.

Test trials were performed to verify instrumentation, fixture design, and procedure on two specimens. Both specimens failed in the outer bolt holes. Therefore, it appeared that the predicted failure stress of 17,000 psi was too low or that combined loads were entering into the gage area due to deflections. Thus, the specimen configuration was inadequate for the test intended. A decision was made to reduce test failure loads by notching the previous specimens as seen in Figure 27. Although test results would not reflect pure shear failures, they would give a relative comparison of thermal damage when tested at room temperature and 220°F.

Data obtained in these tests are tabulated in Table 8. Most of these modified specimens failed along the dashed line shown in Figure 27. Visual damage was present in specimen T11-1 through T11-6 which had been subjected to 600°F thermal shock. Yet, test results from comparable specimens SA4-1 and SA4-2 were nearly identical.

These data may indicate no degradation of residual strength as a result of thermal shock when the specimens were tested at room temperature and 220°F. However, notch effects in the specimen result in combined stresses. Therefore, no conclusions may be reached regarding the residual shear strengths of specimens subjected to prior thermal shocks.

G. COMPRESSION SPECIMEN TESTS

Coated 0.500-inch-wide x 4.400-inch-long compression specimens were cut from 48-ply panels which had been subjected to exposure temperatures of 350°, 400°, 450°. 500°, and 600°F. These exposures were maintained until backside (cool) temperatures stabilized. Ultrasonic examinations of these panels disclosed no evidence of degradation.

Compression testing of these specimens was performed in an Instron test machine with the load applied perpendicular to the ends of the 0° fiber orientation. Figures 28 and 29 show the test fixtures used for testing at 77° and 220°F in the conditioning box.

The resulting data (Table 9) did not conform to an expected pattern. Although past experience has shown considerable scatter in individual specimen data, the average values would fall into predictable patterns. Review of panel exposure histories, postcutting inspections, and test procedures did not show any basis for these erratic results. Therefore, these data are not considered valid for residual compressive strengths.

Table 10 presents the exposure temperatures and times for those panels cut into rail shear and compressive specimens.

H. THERMAL DISCOLORATION MEASUREMENTS

Small 3/4- x 3/4- x 1/4-inch-thick coupons were exposed to high temperatures striking the coated face. A circulating lab oven was used for exposure temperatures of 350°, 400°, 450°, and 500°F. A number of coupons were placed in the oven upon a piece of soft ceramic blanket insulation. These coupons were withdrawn from the heated oven at various times.

The Lindberg furnace was used for exposures at 600°F and up. Each coupon was exposed individually while being held with a pair of tongs. Additional 1200°F exposures of coupons were made to determine which of the materials were the first to exhibit flames. The following observations were made:

(1)	Coating over primer	Flamed at 75 seconds
(2)	Primer only	Flamed at 60 seconds
(3)	Graphite composite only	Flamed at 50 seconds

It appears that the top coating (MIL-C-81773, Type I) and the primer (MIL-P-23377, Type II) inhibit combustion of the AS-1/3501-6 graphite composite degradation products for only a short period of time.

Light reflection measurements were performed using a Leitz Research microscope (Figure 30) which measures reflected light quantitatively. The coupons were hit with a constant-strength light source while the detector was supplied with a constant voltage. The slit was 20 microns in width and used with a 20X objective lens (Air). Readings were first taken on coated coupons that had not been exposed to high temperatures. These were considered as standards. Readings were then taken on coupons that were subjected to the various high temperatures for different periods of time. These readings were then converted to a percent of reflected light when compared to the standards. (See Table 11.) The results of these exposures are shown in Figure 31.

Review of top coating color changes as a result of thermal exposure on the test coupons shows that, at exposures of 600°F and above, irreversible first-ply damage can be expected with sufficient exposure times. The damage threshold occurs when the top coat color changes to a mauve (pale violet) shading and as the percent of reflected light drops to approximately 80% of the unexposed coat. Color changes from light gull grey through tan through brown do not indicate first-ply damage unless there is evidence of the mauve shading. This mauve color first occurs during 600°F exposures and is found during coating color changes at the higher temperature exposures.

A plot of predicted threshold damage (time/temperature) is presented in Figure 32.

SECTION III

CONCLUSIONS

Strength degradation caused by thermal shock while the laminate is under a sustained 2500 μ in./in. strain (design limit) is related to the type of load imposed. During shock temperature up to 1200°F, the exposed durations for panels carrying sustained compression strain during these tests were significantly longer than demonstrated by flex panels undergoing the same exposure temperatures and strain (measured on tension side). Panels subjected to sustained flex loading also failed at lower exposure temperatures than panels undergoing sustained compression. Flexural load-carrying ability (2500 μ in./in. strain) for 1.6 minutes was demonstrated during a 1800°F exposure.

Delaminations may occur initially in panels undergoing 600°F thermal shock. NDT examinations showed one of three panels exposed to 600°F to have an internal ply delamination.

At exposure temperatures of 800°F and above, substantial structural degradation of major proportions was very evident. Postexposure examination showed gross delaminations through the panel thickness, plies adjacent to the shocked surface falling away, resin charring, and panels warping. High exposure temperatures caused outgassing and ablation affects which momentarily reduce heat transfer through the panel.

Top-coating color changes begin at 400°F with a pale tan and progress through a mauve, deep tan, brown, and black at the high exposures. Color changes of the top coating below 600°F exposure are different than those above 600°F. A mauve color indicates that the degradation threshold of the first ply of the composite has been reached.

Neither visual examinations nor panel thickness measurements are positive in determining if degradation (delamination) has occurred. Ultrasonic examinations will confirm that a delamination has occurred but will not indicate whether the resin has been permanently damaged and/or the laminate properties reduced.

SECTION IV

RECOMMENDATIONS

The effort in this study was performed on dry AS-1/3501-6 composite specimens and panels. Additional investigations should be made on this composite which contains absorbed moisture in excess of 1.0% by weight.

A means should be found to determine when structural damage begins during thermal shock. Use of acoustic devices may furnish this type of data.

Additional test methods should be employed to determine degree of permanent resin matrix damage thresholds. Transverse tensile tests or a special short-beam shear test after thermal shock would provide meaningful data.

TABLE 1. AS-1/3501-6 COUPON TEST DATA AT 220°F

Property	$\overline{x}$	σ	C_V%	n
E_{11t}, msi	20.14	0.9223 msi	4.58	18
E_{11c}, msi	19.27	1.3241 msi	6.84	16
E_{22t}, msi	1.376	0.1524 msi	11.08	16
E_{22c}, msi	Assume E_{22c} = E_{22t}			
G_{12}, msi (tangent)	0.620	0.0391 msi	6.30	7
G_{12}, msi (secant)	0.330	0.0346 msi	10.50	7
ν_{12}	0.309	0.0291	9.40	18
ϵ_{11t}, in./in.	0.01215	0.01482	12.20	18
ϵ_{11c}, in./in.	0.00966	0.00102	10.53	14
ϵ_{22t}, in./in.	0.00520	0.00094	17.79	16
ϵ_{22t}, in./in.	0.01870	0.00263	14.06	15
ϵ_{12}, in./in. (tangent)	0.00767	0.00077	10.0	7
ϵ_{12}, in./in. (secant)	0.01950	0.0098	5.0	7
σ_{11t}, ksi	243.96	25.21	10.33	18
σ_{11c}, ksi	192.42	14.618	7.60	12
σ_{22t}, ksi	7.17	0.8523	11.89	18
σ_{22c}, ksi	25.73	--	--	--
σ_{SBS}, ksi	13.45	0.719	0.05	18

TABLE 2. TEMPERATURE GRADIENTS DURING PANEL EXPOSURES

Exposure Temperature (Total Exposure Time)	Temperature Gradients	Elapsed Time (min)			
		T 12	T 24	T 36	T 48
350°F	200	---	6.0	7.0	8.0
	250	---	16.0	25.0	---
	T max	---	25.0 (256°)	35.0 (253°F)	30.0 (235°F)
400°F	200	---	4.5	5.0	5.0
	250	---	8.0	9.0	7.5
	300	---	---	70.0	---
	T max	---	75 (291°F)	70 (300°F)	75 (285°F)
450°F	200	4.5	4.5	5.0	7.0
	250	9.5	7.5	8.0	25.0
	300	19.0	15.0	16.0	38.0
	T max	55 (297°F)	60 (340°F)	60 (345°F)	65 (302°F)
500°F	200	4.5	5.0	4.5	4.5
	250	5.5	8.5	6.5	7.5
	300	8.5	15.5	11.5	14.0
	350	21.0	39.0	---	8.0
	400	45.0	---	---	---
	T max	90 (415°F)	60.0 (356°F)	90.0 (338°F)	90.0 (358°F)

--- Indicates faulty thermocouple

TABLE 2. TEMPERATURE GRADIENTS DURING PANEL EXPOSURES (Cont)

Exposure Temperature (Total Exposure Time)	Temperature Gradients	Elapsed Time (min)			
		T 12	T 24	T 36	T 48
600°F	200	2.25	1.75	1.0	2.5
	250	3.5	3.0	2.0	3.5
	300	5.5	5.5	4.0	6.0
	350	9.5	9.0	7.5	11.0
	400	25.0	18.0	18.0	31.0
	T max	41.0 (412°F)	38.0 (428°F)	40.0 (420°F)	38.0 (410°F)
800°F	200	0.5	0.5	0.5	1.00
	250	1.0	0.75	0.75	2.50
	300	1.5	1.00	1.00	3.50
	350	2.20	1.75	1.90	4.20
	400	2.75	2.3	2.75	5.8
	450	4.00	3.0	3.8	9.5
	500	5.50	4.0	5.00	15.5
	600	---	---	29.0	---
	T max	10.0 (530°F)	7.5 (562°F)	35.0 (605°F)	50.0 (550°F)
1000°F	200	0.25	0.50	0.50	0.50
	250	0.85	0.90	0.85	0.65
	300	1.40	1.40	1.50	1.10
	350	1.90	1.80	1.80	1.70
	400	2.60	2.30	2.50	2.10
	450	3.25	2.95	3.00	2.60
	500	3.90	3.50	3.70	3.10

TABLE 2. TEMPERATURE GRADIENTS DURING PANEL EXPOSURES (Cont)

Exposure Temperature (Total Exposure Time)	Temperature Gradients	Elapsed Time (min)			
		T 12	T 24	T 36	T 48
1000°F (Cont)	550	4.75	4.25	4.45	3.75
	600	5.90	5.10	5.75	5.60
	650	7.50	9.00	---	---
	700	13.0	21.50	---	---
	750	21.5	---	---	---
	T max	25.0 (763°F)	25.0 (728°F)	25.0 (633°F)	5.60 (600°F)
1800°F	200	---	0.40	0.60	0.50
	250	---	0.60	0.77	0.65
	300	---	0.72	0.95	0.80
	350	---	0.90	1.12	0.92
	400	---	1.08	1.35	1.11
	450	---	1.25	1.55	1.35
	500	---	1.48	1.82	1.65
	550	---	1.75	2.25	2.70
	600	---	2.10	3.50	---
	650	---	3.50	5.35	---
	700	---	6.20	7.25	---
	750	---	7.00	9.70	---
	800	---	8.00	---	---
	850	---	9.00	---	---
	900	---	10.00	---	---
	T max	---	10.0 (900°F)	10.0 (755°F)	10.0 (580°F)

TABLE 3. PANEL WEIGHT LOSSES DUE TO THERMAL EXPOSURE

Exposure Temperature (°F)	Total Exposure Time (min)	Weight Loss (%)
350	120	0.015
400	120	0.027
450	90	0.029
500	90	0.107
600	90	0.265
800	50	3.155
1000	25	6.435
1400	10	8.534
1800	10	11.071

TABLE 4. PANELS EXPOSED WITH EMBEDDED THERMOCOUPLES

Panel No.	Exposure Temperature (°F)	Exposure Time (min)
T-4	350	120
T-5	400	120
T-4	450	90
T-5, T-6, T-9	500	90
T-11	600	90
T-12	800	50
T-13	1000	25
T-5	1400	10
T-14	1800	10

Note that some panels received multiple exposures when prior exposures did not inflict damage.

TABLE 5. PANEL EXAMINATION RESULTS

Exposure Temperature (°F)	Total Exposure Time (min)	Ultrasonic Results	Change in Appearance of Coated Surface
350	120	No change	None
400	120	No change	Color darkened very slightly
450	90	No change	Very light tan
500	90	No change	Light tan
600	90	Delaminated	Light brown
800	50	Delaminated	Dark green to black, major disintegration
1000	25	Delaminated	Black with grey residue, major disintegration
1400	10	Delaminated	Black, major disintegration
1800	10	Delaminated	Black, major disintegration

TABLE 6. SUSTAINED COMPRESSION TEST RESULTS

Thermal Shock Temp (°F)	Panel No.	Exposure Time (min)	Cool Side Temp °F at End of Test	Maximum Strain Measured (μ/in./in.)	Results(2)
350	C-5	40	202	2540	Carried stress of 24,470 psi
400	C-5	40	229	2500	Carried stress of 24,470 psi
450	C-5	40	250	2575	Carried stress of 25,300 psi
500	C-5	55	271	2550	Carried stress of (1) psi
550	C-4	125	326	2225	Carried stress of 25 psi
600	C-2	73	314	2550	Failed under load
800	C-14	18	205	3900	Failed under load
1200	C-16	3	360	3950	Failed under load
1600	C-12	3	510	2530	Failed under load
1800	C-1	2	125	1650	Failed under load

(1) Taken to failure load of 151,000 pounds after 55 minutes at 500°F, S_1 = 6323 (hot); S_2 = 8653; P/A = 63,236 psi

(2) Two unexposed specimens were tested in this set up at R.T. with average failure stress of 82,808 psi.

TABLE 7. FOUR-POINT FLEX TEST DATA

Panel No.	Exposure Test Temp (°F)	Soecimen Thickness (in.)	Specimen Width (in.)	Initial Loads (lb)	Peak Load (lb)	Maximum Surface Temp (°F)	Exposure Time (min)	Results
4P-1	350	0.280	1.494	375	730	370	42.0	Maintained strain loading at 37,395 psi
4P-1	400	0.280	1.494	750(1)	940	423	41.0	Maintained strain loading at 48,150 psi
4P-6	450	0.280	2.498	380	1120	453	16.0	Failed at 57,220 psi
4P-5	500	0.280	1.494	370	1050	515	10.0	Failed at 53,786 psi
4P-4	600	0.278	1.493	405	1080	530	10.5	Failed at 56,160 psi
4P-3	800	0.278	1.495	390	1010	620	5.5	Failed at 52,450 psi
4P-2	1200	0.283	1.495	420	1145	770	3.0	Delamination failure at 52,380 psi
4P-7	1600	0.277	1.497	370	860	890	3.5	Softening failure at 44,925 psi
4P-8	1800	0.276	1.495	365	790	860	2.5	Softening failure at 41,620 psi
1(2)	RT	0.282	1.500	---	2060	77	---	Avg failure at 98,390 psi
2(2)	RT	0.286	1.500	---	2050	77	---	
3(2)	RT	0.280	1.500	---	1800	77	---	

(1) Continuation of 350°F test
(2) No thermal exposure or instrumentation. For baseline information only
(3) Span to thickness ratio: Design 32/1; Actual 28.571

TABLE 8. RAIL SHEAR SPECIMEN TEST RESULTS

Specimen Exposure Temperature (°F)	Test Temp (°F)	Avg Failure Stress (psi) / CV%	Avg Recorded Strain (%) / CV%
350	77	28075 / CV = 11.0	0.635 / CV = 22.0
	220	27290 / CV = 3.5	0.510 / ---
400	77	28742 / CV = 5.2	0.680 / CV = 13.3
	220	27932 / CV = 8.6	0.477 / CV = 17.9
450	77	30437 / CV = 11.4	0.653 / CV = 16.1
	220	32018 / CV = 4.8	0.491 / CV = 29.5
500	77	28758 / CV = 10.9	0.640 / CV = 11.9
	220	30251 / CV = 8.2	0.653 / CV = 5.5
600	77	32075 / CV = 1.9	---
	220	29270 / CV = 13.2	0.642 / CV = 26.8

$$CV = \frac{S_x}{\bar{x}} \times 100$$

TABLE 9. COMPRESSION SPECIMEN TEST DATA

Exposure Temp (°F)	Test Temp (°F)	Avg Failure Stress (psi) / Coefficient of Variation (%)
350	77	90854 / CV = 14.7
	225	71744 / CV = 14.7
400	77	64159 / CV = 4.7
	220	53085 / CV = 6.8
450	77	72256 / CV = 9.5
	220	57067 / CV = 15.4
500	77	78840 / CV = 7.5
	220	64142 / CV = 27.3
600	77	74617 / CV = 14.1
	220	61937 / CV = 2.8

$$CV = \frac{S_x}{\bar{x}} \times 100$$

TABLE 10. EXPOSED PANELS FOR RESIDUAL STRENGTH RAIL SHEAR AND COMPRESSION SPECIMENS

Panel No.	Exposure Temperature (°F)	Exposure Time (min)	% Wt. Loss	No. of Specimens	
				R.S.	Comp.
S-C1	350	60	-	2	8
S-B2	350	60	-	6	-
S-C2	400	60	-	6	-
S-B1	400	60	-	2	8
T-4	450	210	0.03	6	-
S-A3	450	60	0.08	2	8
T-6	500	120	0.01	6	-
T-7	500	120	0.01	2	8
T-11	600	-	-	6	-
S-A4	600	60	0.17	2	8

TABLE 11. PERCENT REFLECTED LIGHT VERSUS TEMPERATURE/TIME EXPOSURE

350°F	Exposure time, hr	0	2	4	6	8	12	27							
	% light reflected	100	97.9	96.7	95.7	95.3	93.4	88.6							
400°F	Exposure time, min	0	15	30	60	90	120	180	240						
	% light reflected	100	102	102	97.3	100.5	95.8	95.0	92.0						
450°F	Exposure time, min	0	10	15	20	30	45	60	80	100	120	150	180	240	
	% light reflected	100	100.7	99.8	98.6	98.0	95.4	93.1	91.6	90.3	88.4	86.8	84.8	78.6	
500°F	Exposure time, min	0	5	8	10	15	30	45	60	75	90	105	120		
	% light reflected	100	100.6	96.4	95.4	89.6	84.5	80.3	78.8	75.7	73.0	67.7			
600°F	Exposure time, min	0	3	4.5	6	7	8	9	10.5(1)						
800°F	Exposure time, min	0	2	2.5	3(1)	4	4.5								
	% light reflected	100	98.7	84.5	73.8	44.1	34.9	26.7							
1200°F	Exposure time, sec	0	2	5	10	15	20	25	30	35	40	45	50 (2,3)	55	60
	% light reflected	100	99.2	100.5	100.6	93.5	93.9	97.3	96.0	83.6	60.5	6.29	27.4	25.6	30.3
1600°F	Exposure time, sec	0	4	5	6	8	10(2)	12	14(3)						
	% light reflected	100	98.6	92.4	95.7	94.0	83.0	26.3	27.1						
1800°F	Exposure time, sec	0	1	2	3	4(2)	5	6	8	10(3)					
	% light reflected	100	101.3	98.7	99.1	67.5	30.9	25.0	33.2	34.2					

(1) Started smoking
(2) Flame appeared
(3) Delaminated

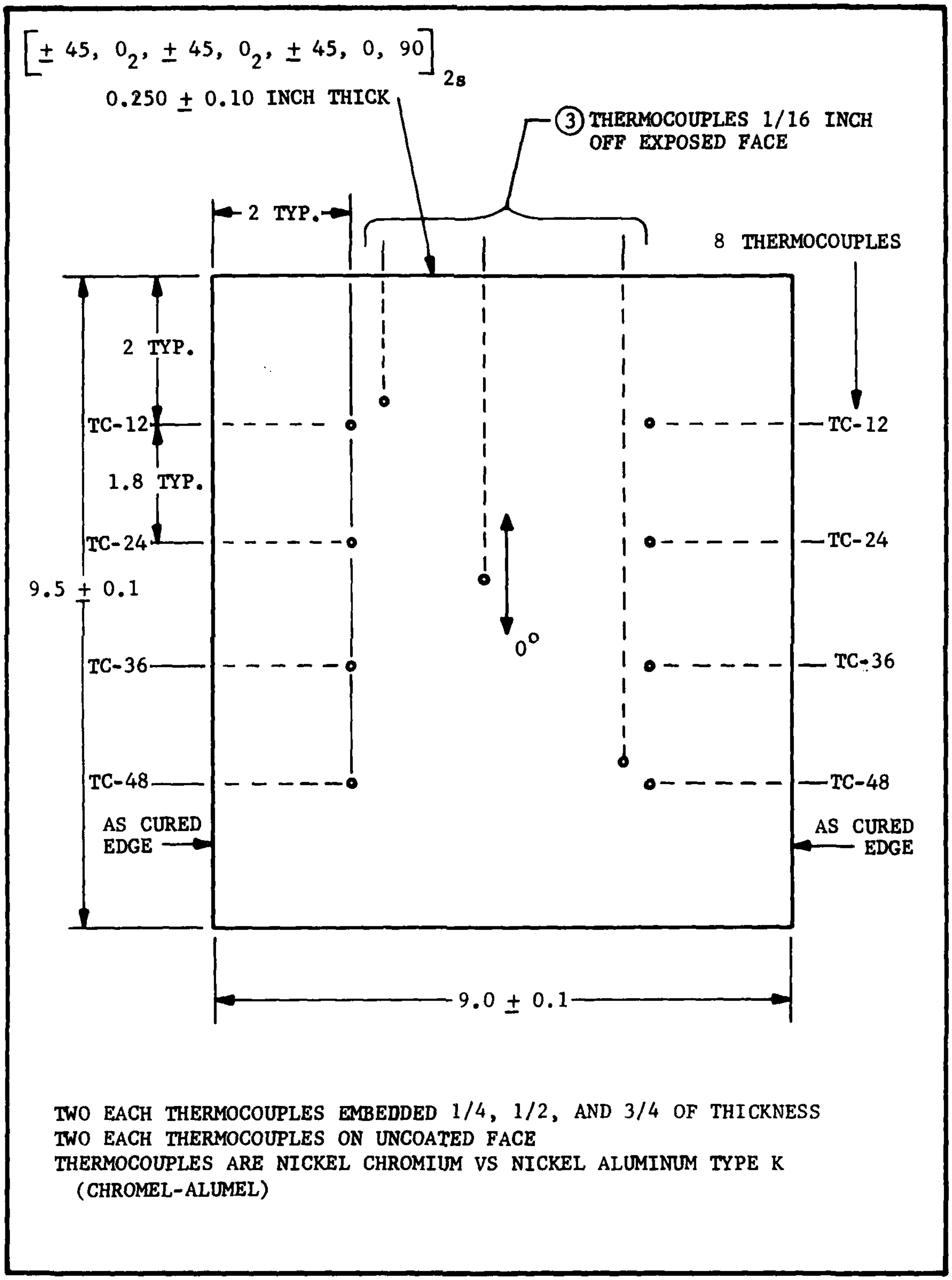


Figure 1. Thermocouple Arrangement for Transit Measurements

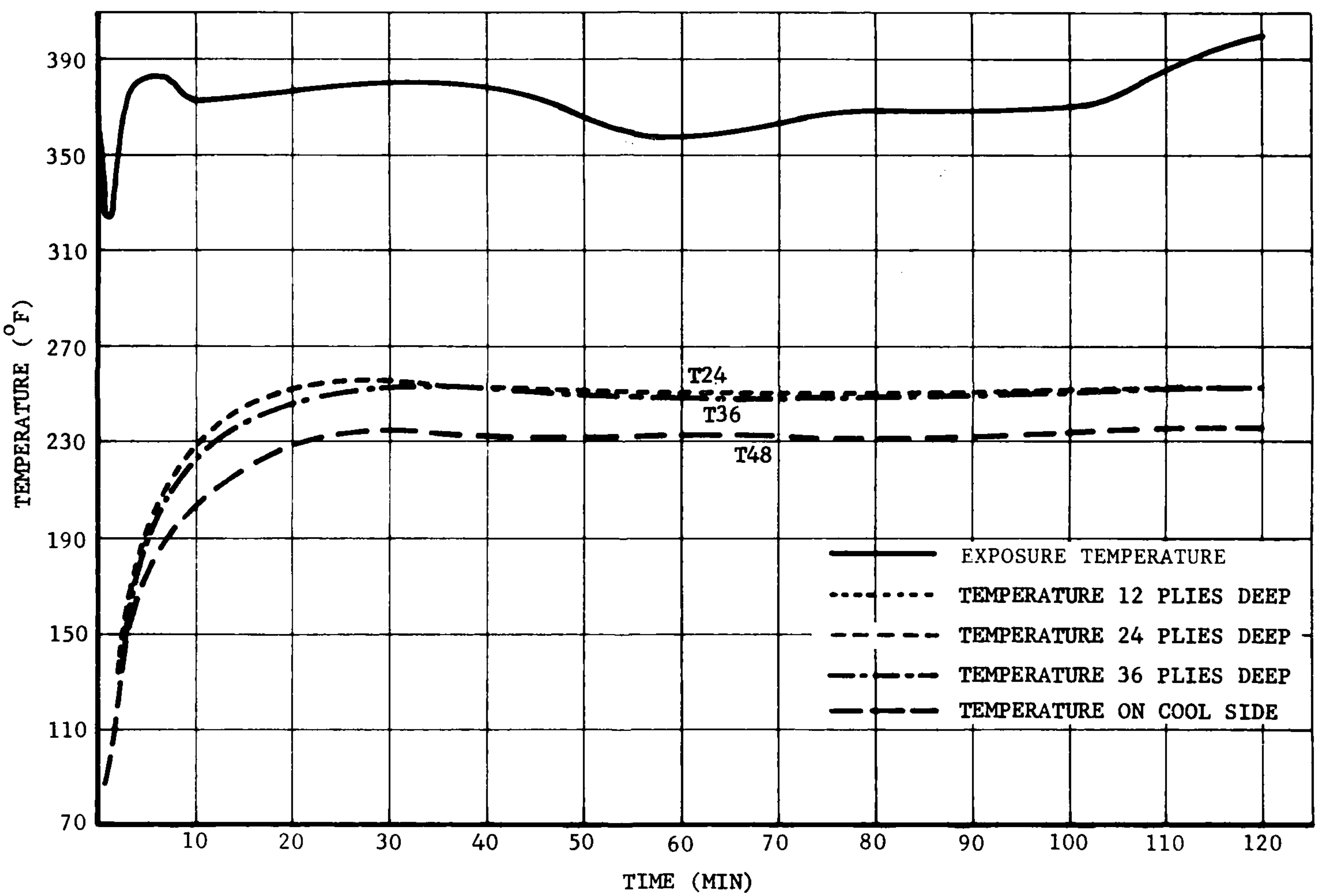


Figure 2. 350^{o}F Exposure Test

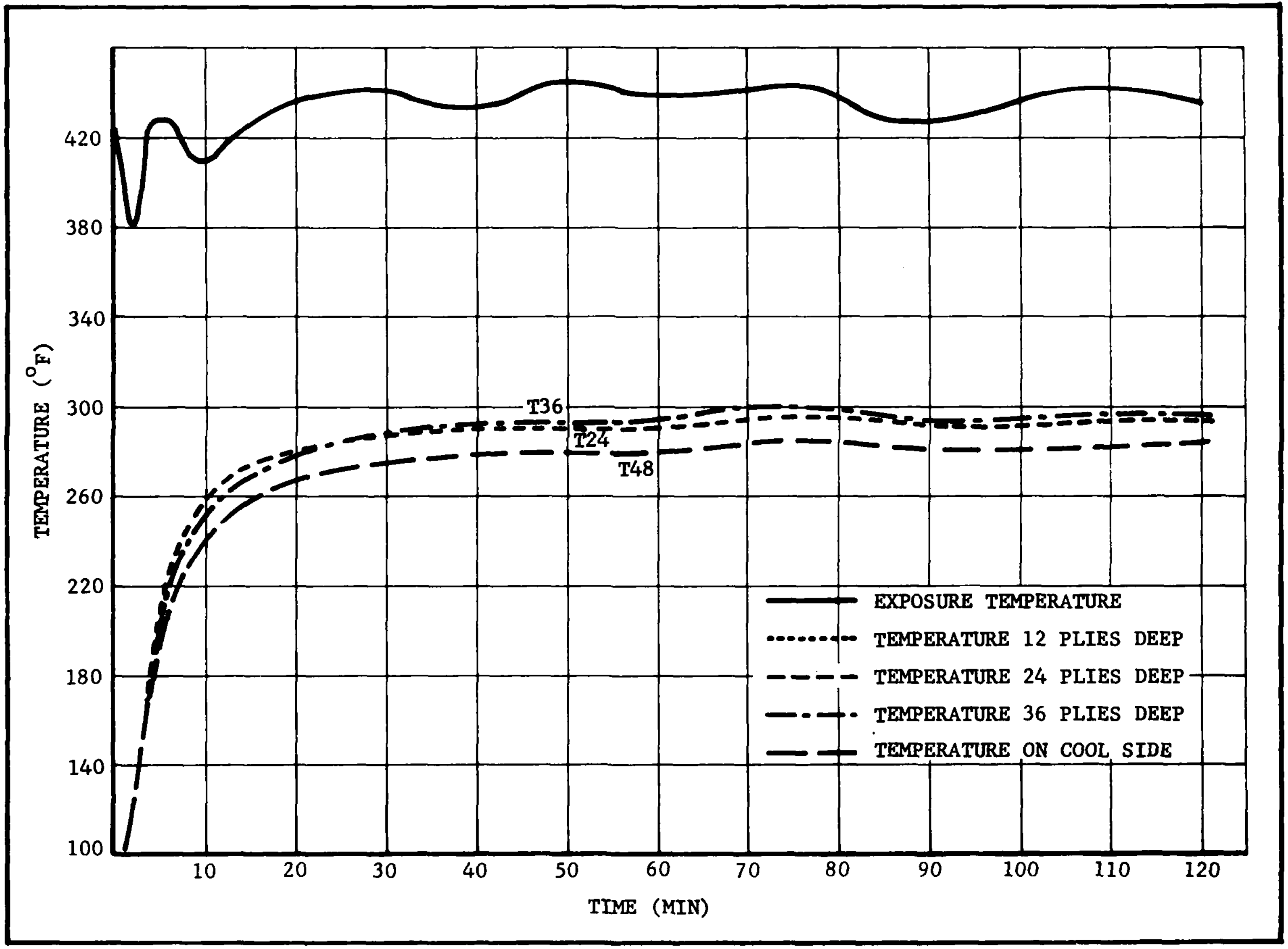


Figure 3. 400°F Exposure Test

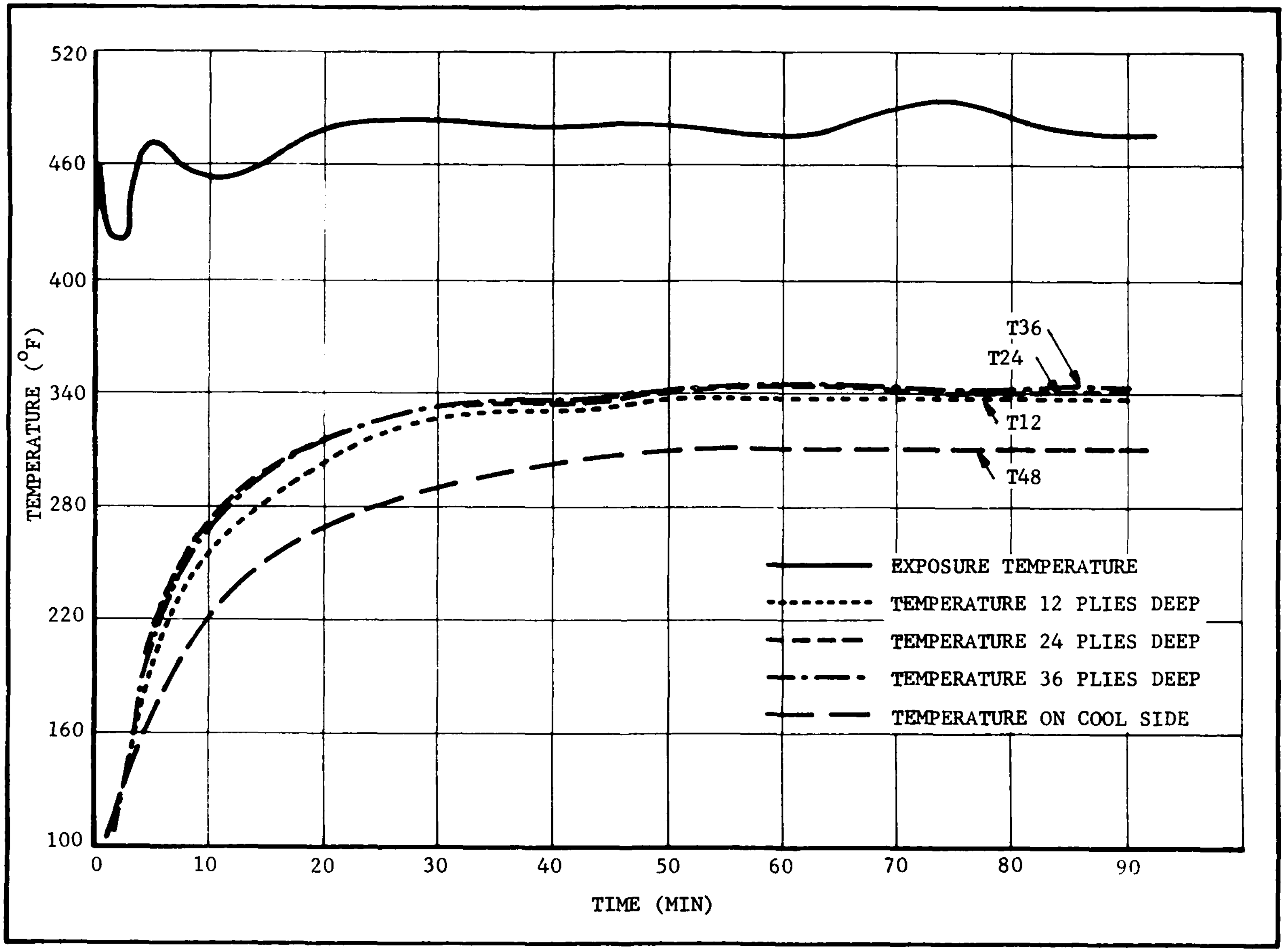


Figure 4. 450°F Exposure Test

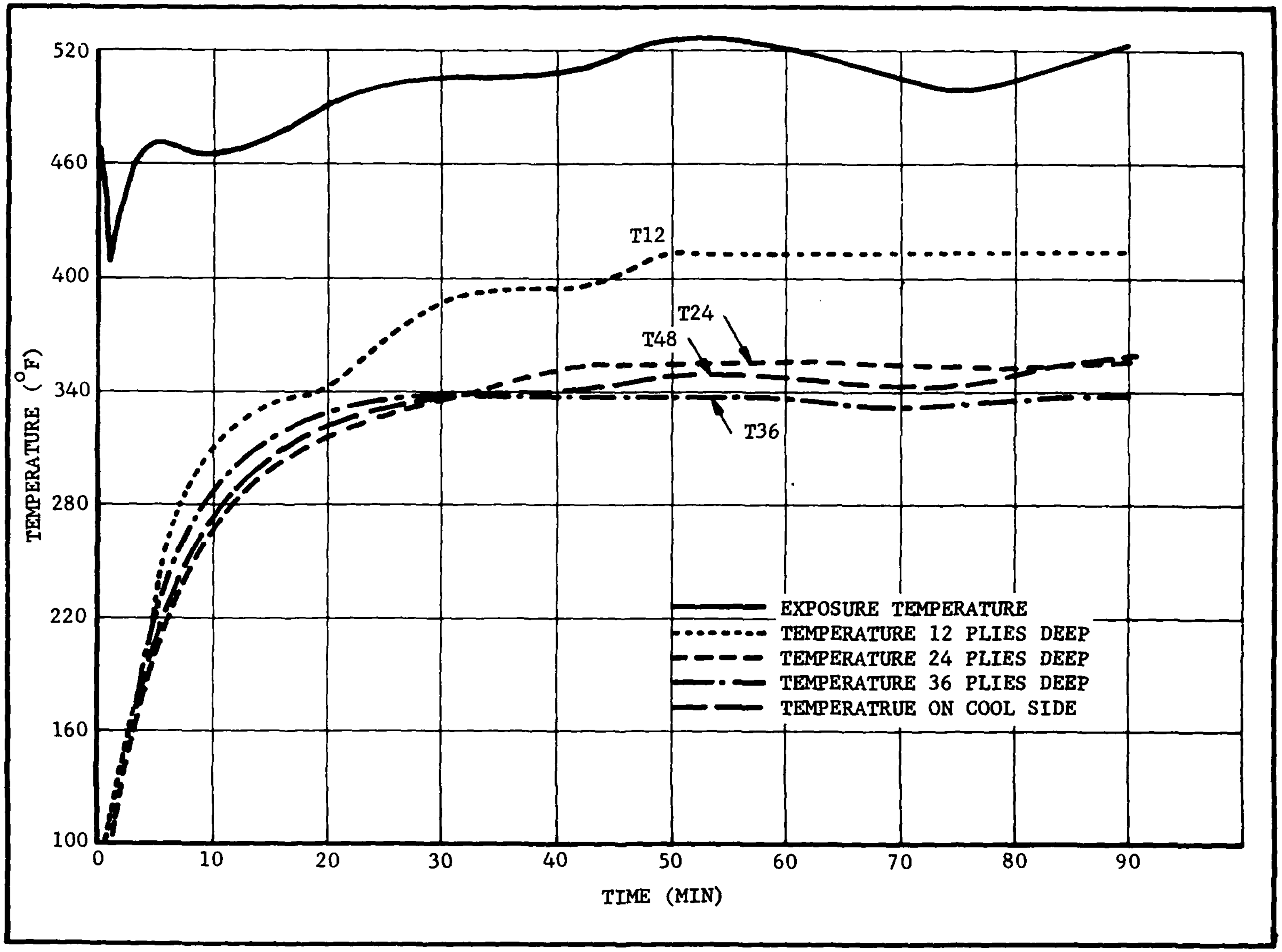


Figure 5. 500°F Exposure Test

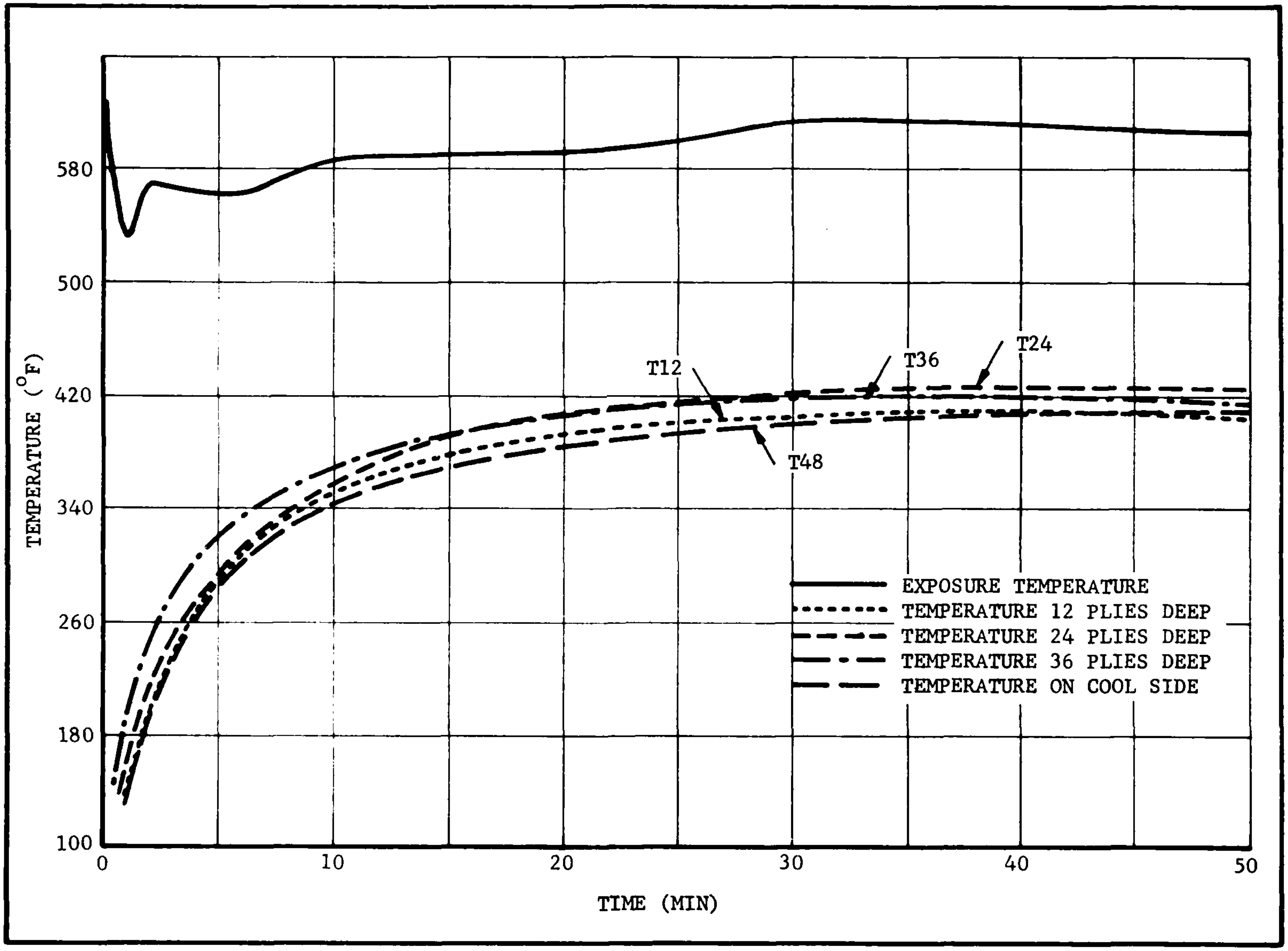


Figure 6. 600°F Exposure Test

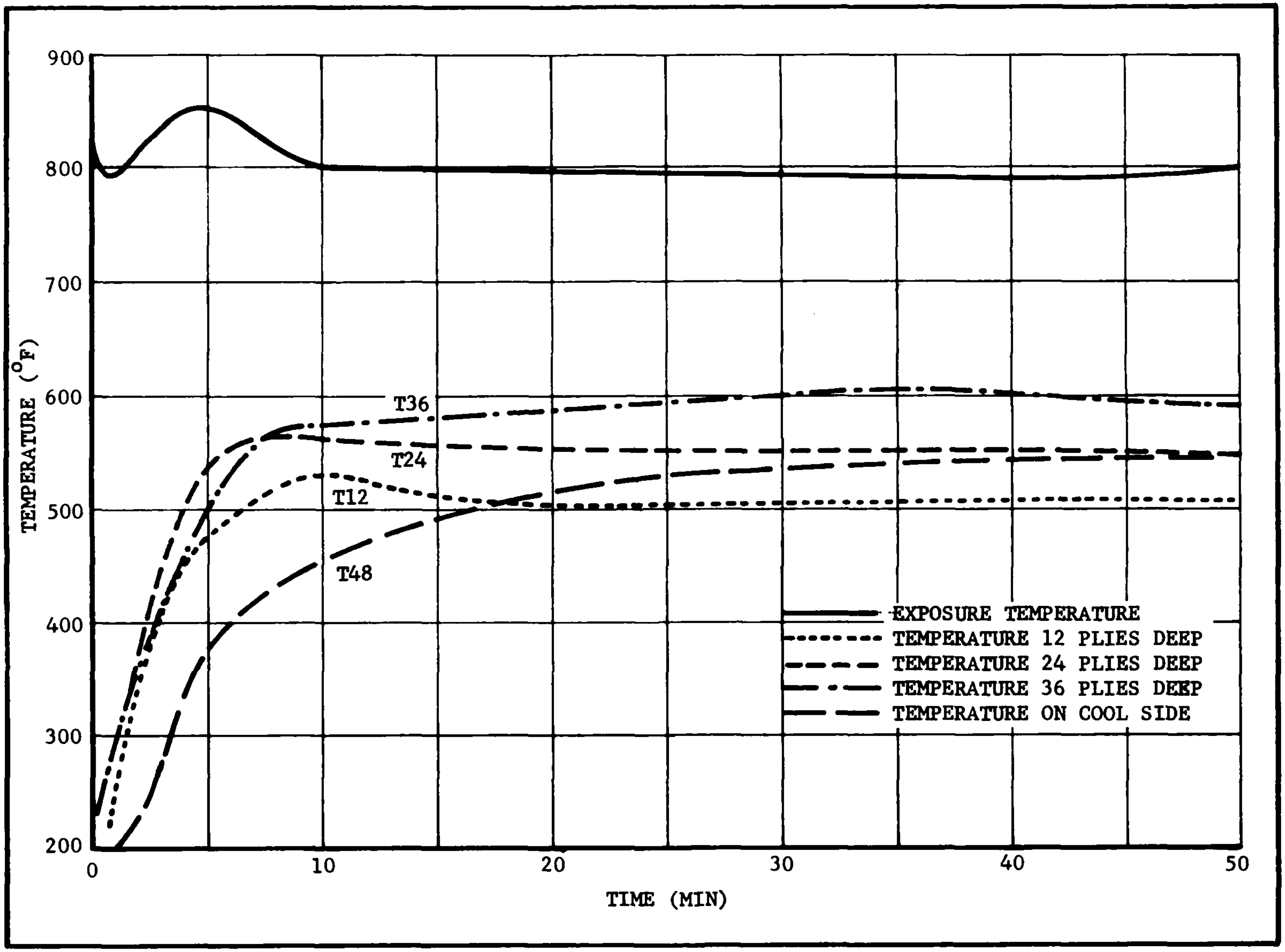


Figure 7. 800°F Exposure Test

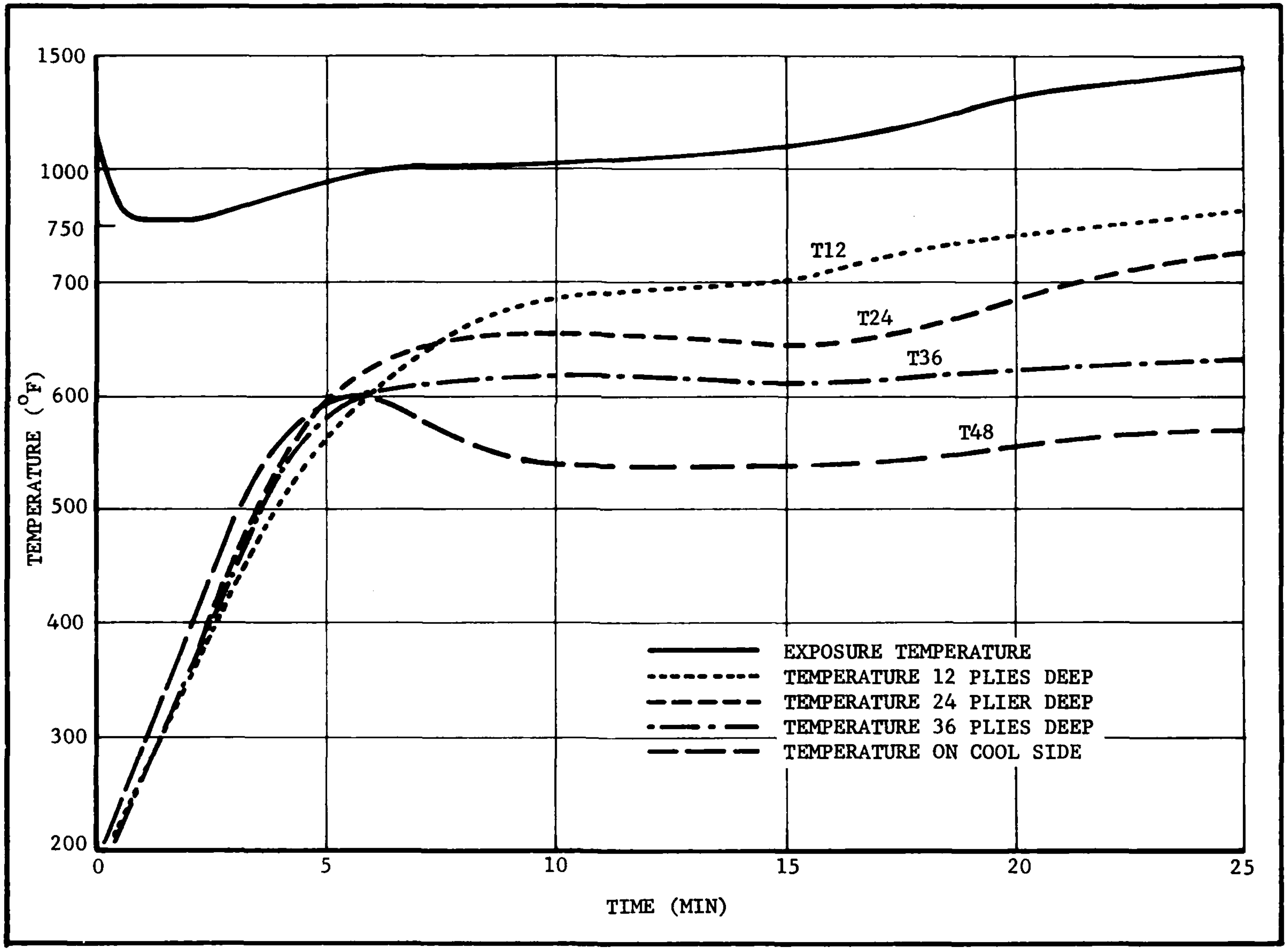


Figure 8. 1000° Exposure Test

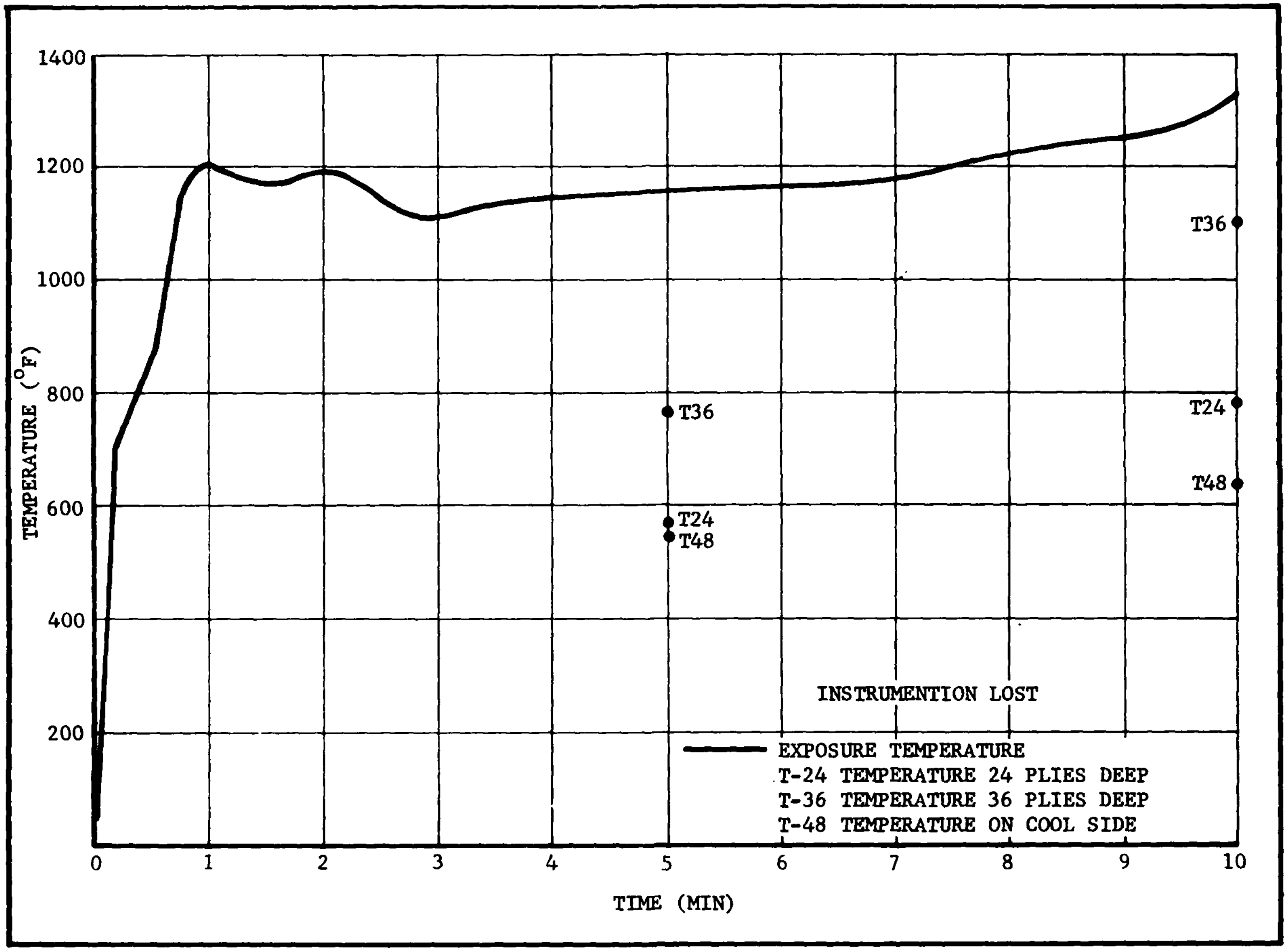


Figure 9. 1400°F Exposure Test

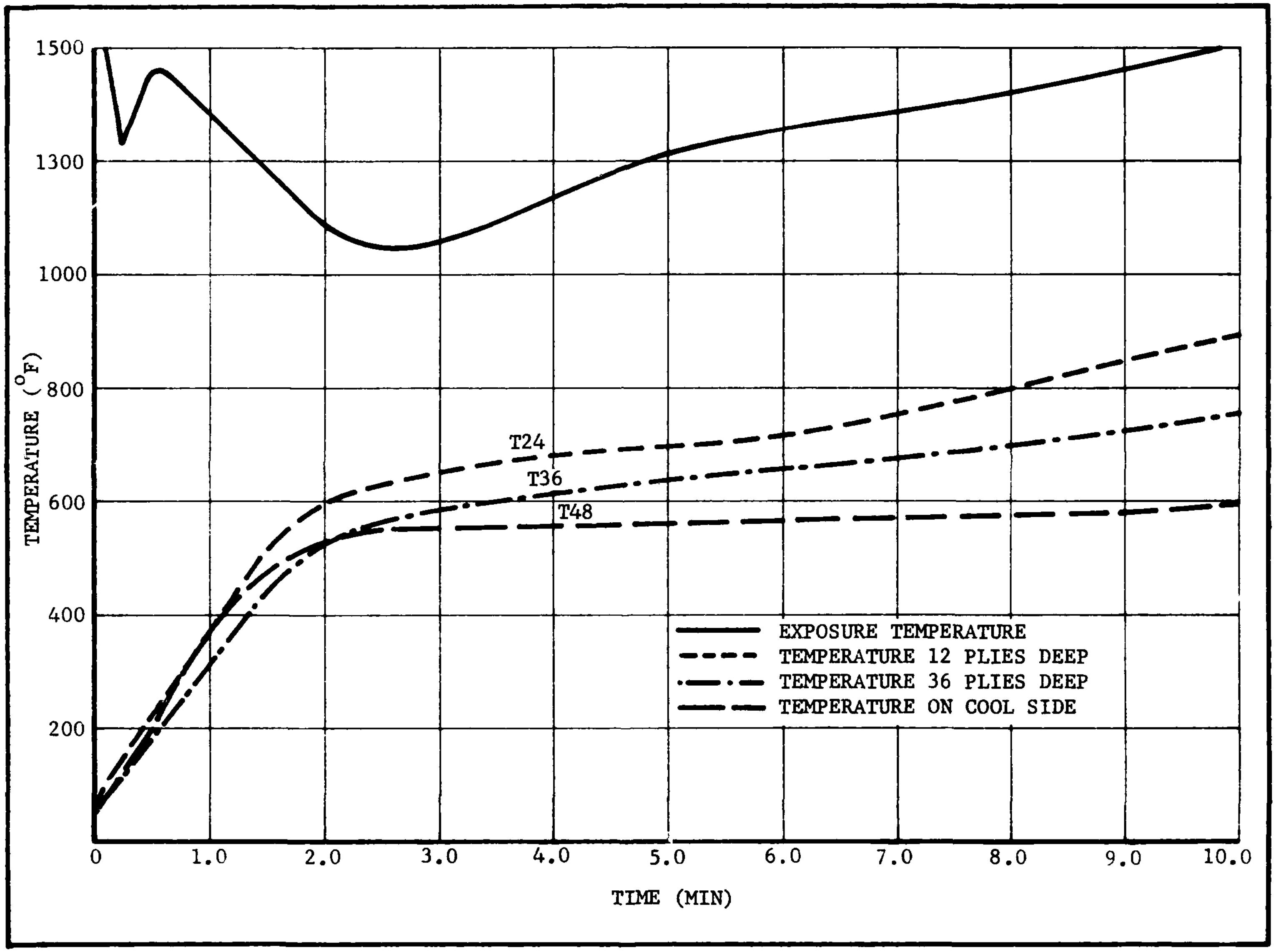


Figure 10. 1800°F Exposure Test

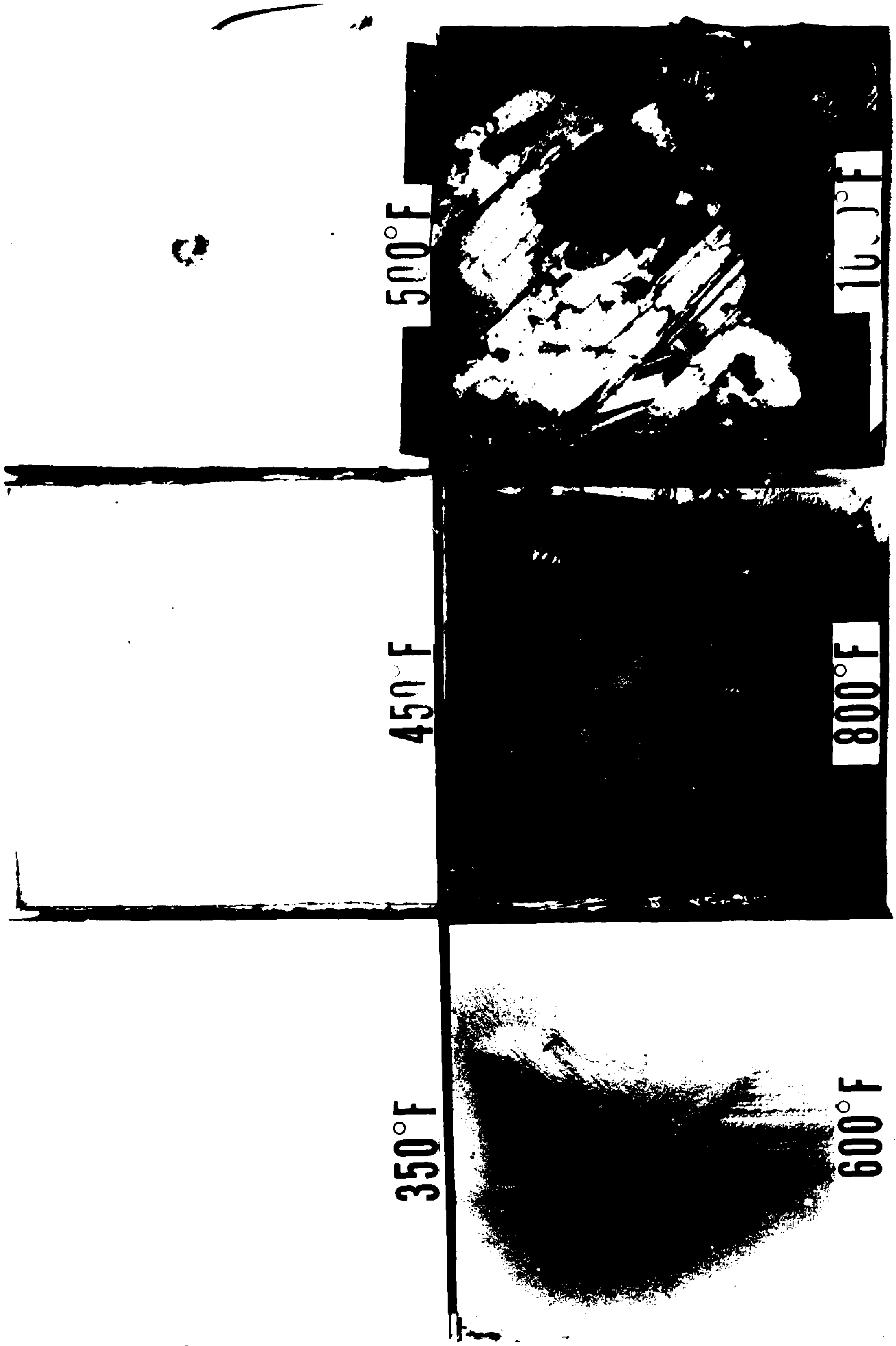


Figure 11. Effects of Thermal Shock

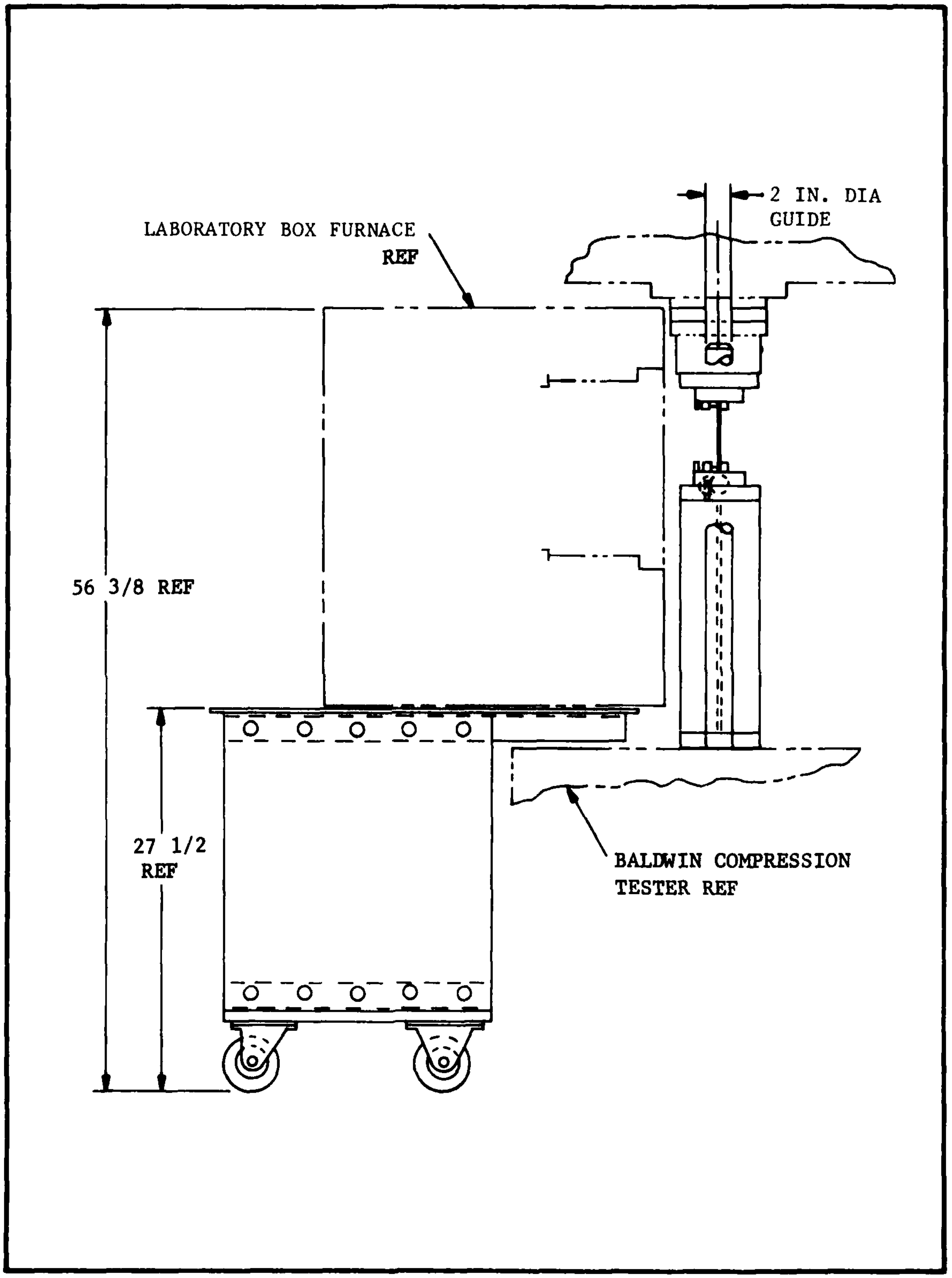


Figure 12. Panel Compression Load Test Equipment Arrangement

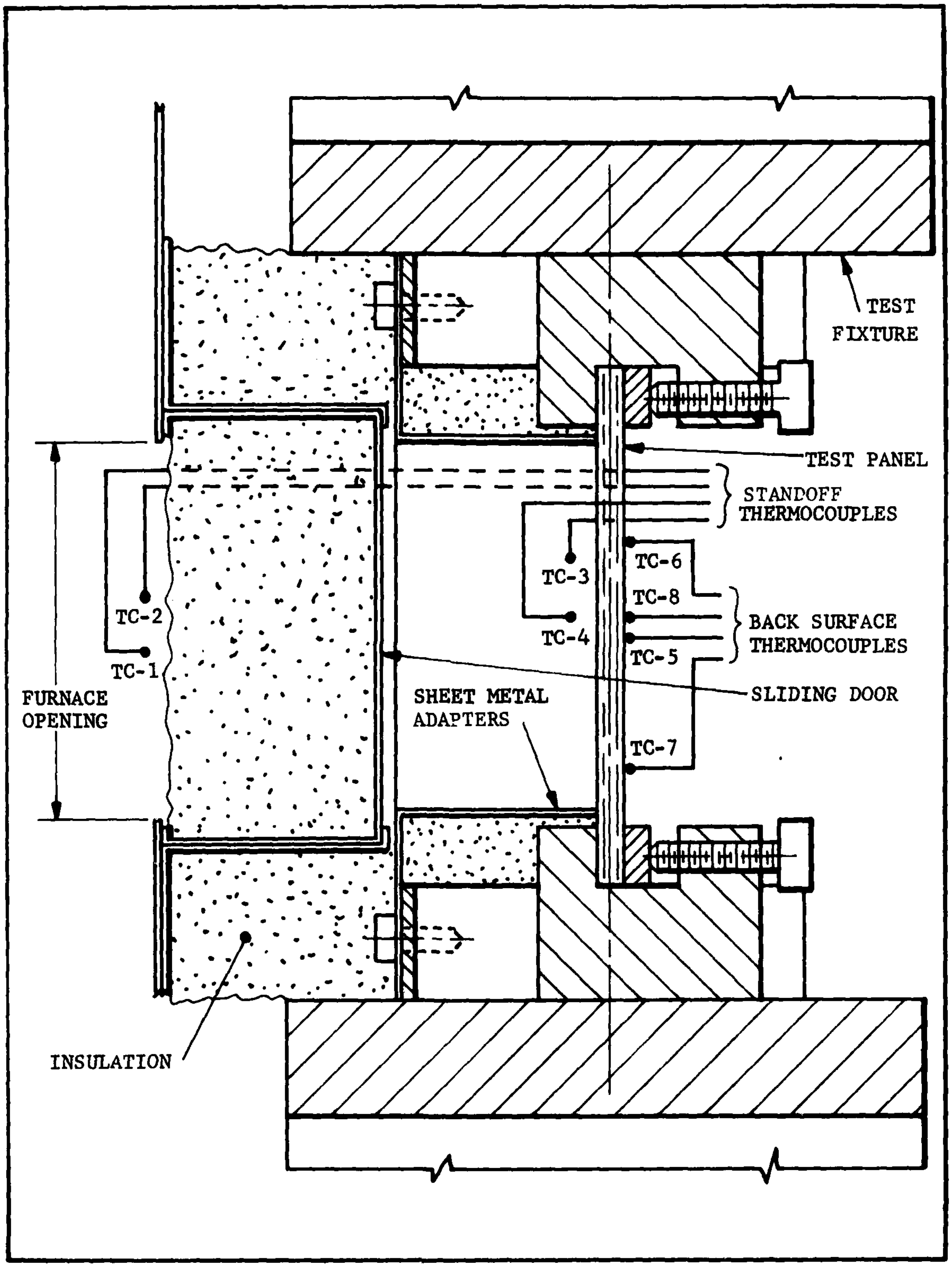


Figure 13. Panel Test Arrangement Details

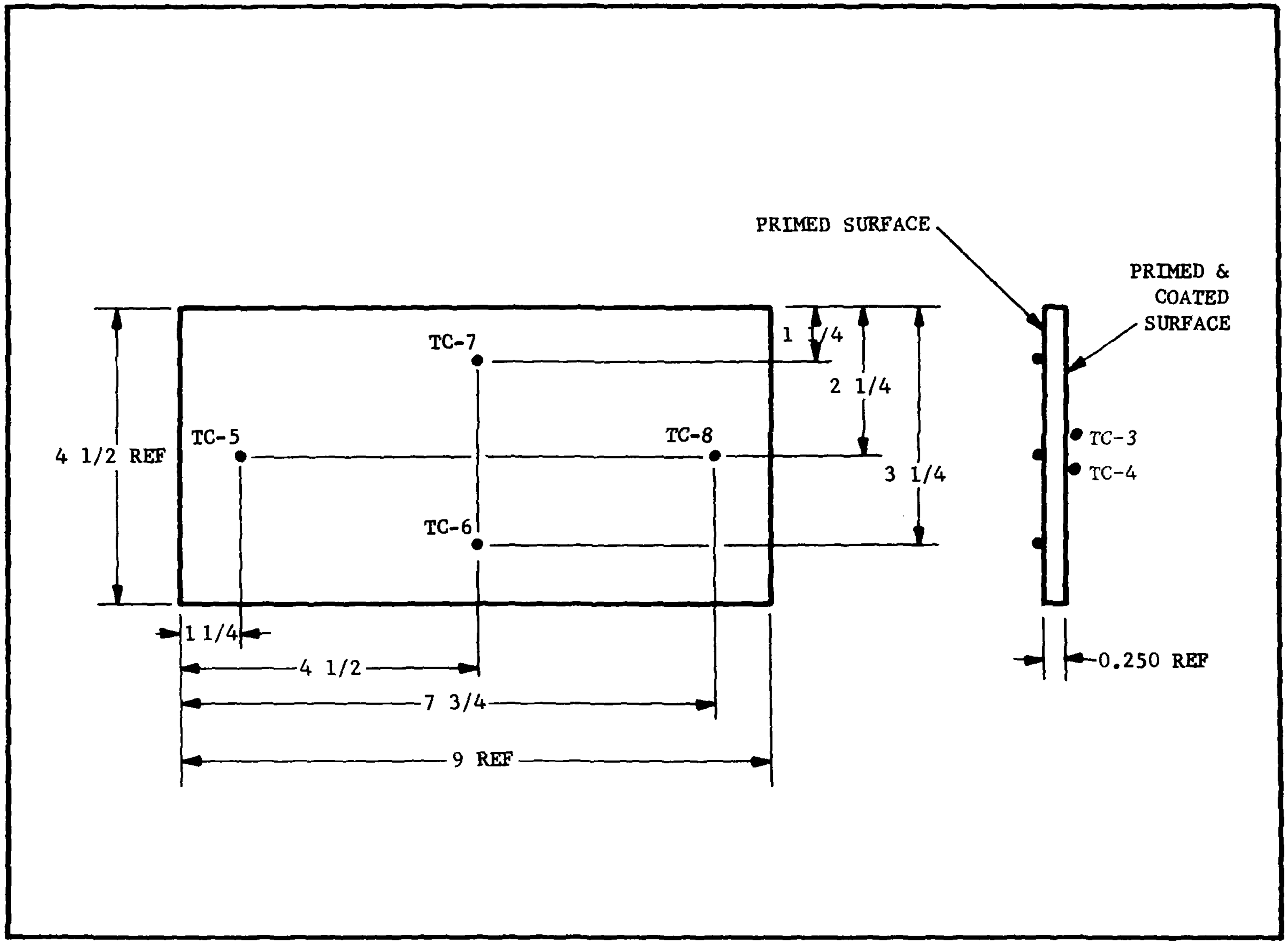
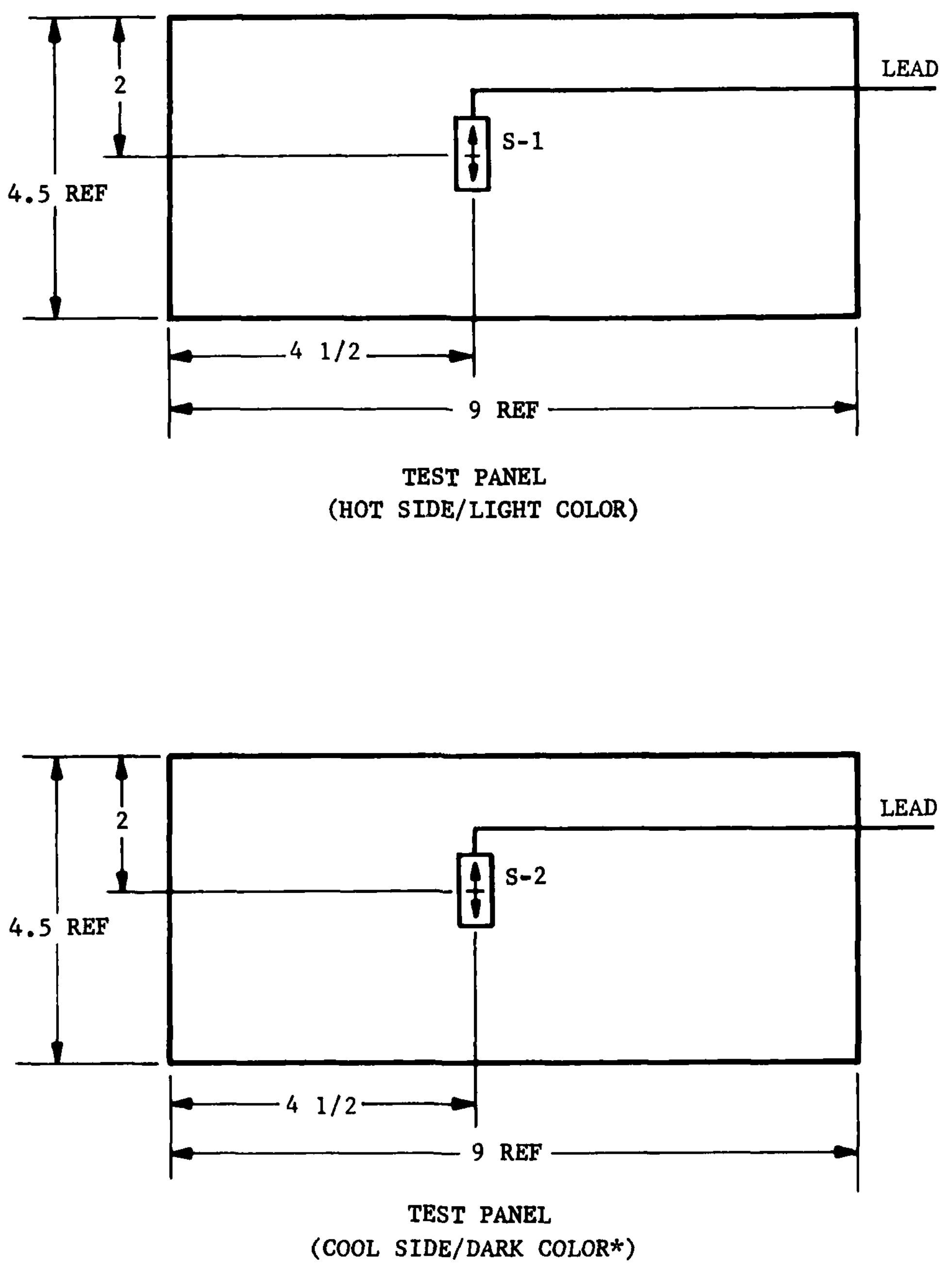


Figure 14. Compression Load Panel Thermocouple Locations



*LOCATION OF S-2 MUST BE OFFSET FROM S-1

NOTE: BOND GAGES WITH BLH EPY-600 AND CURE AT 220°F FOR ONE HOUR

Figure 15. Compression Load Panel Strain Gage Locations

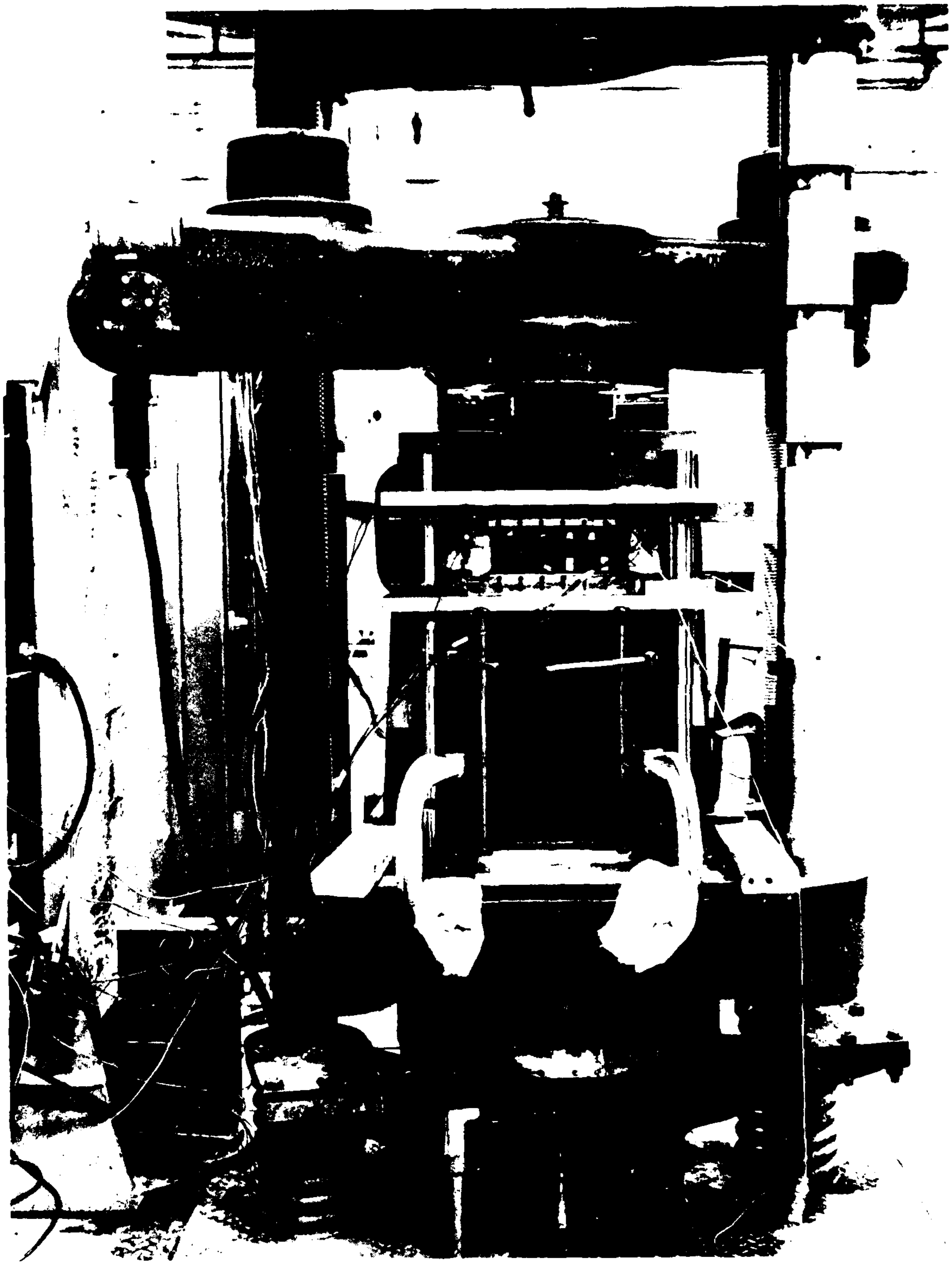


Figure 16. Sustained Compression Thermal Shock Test Setup

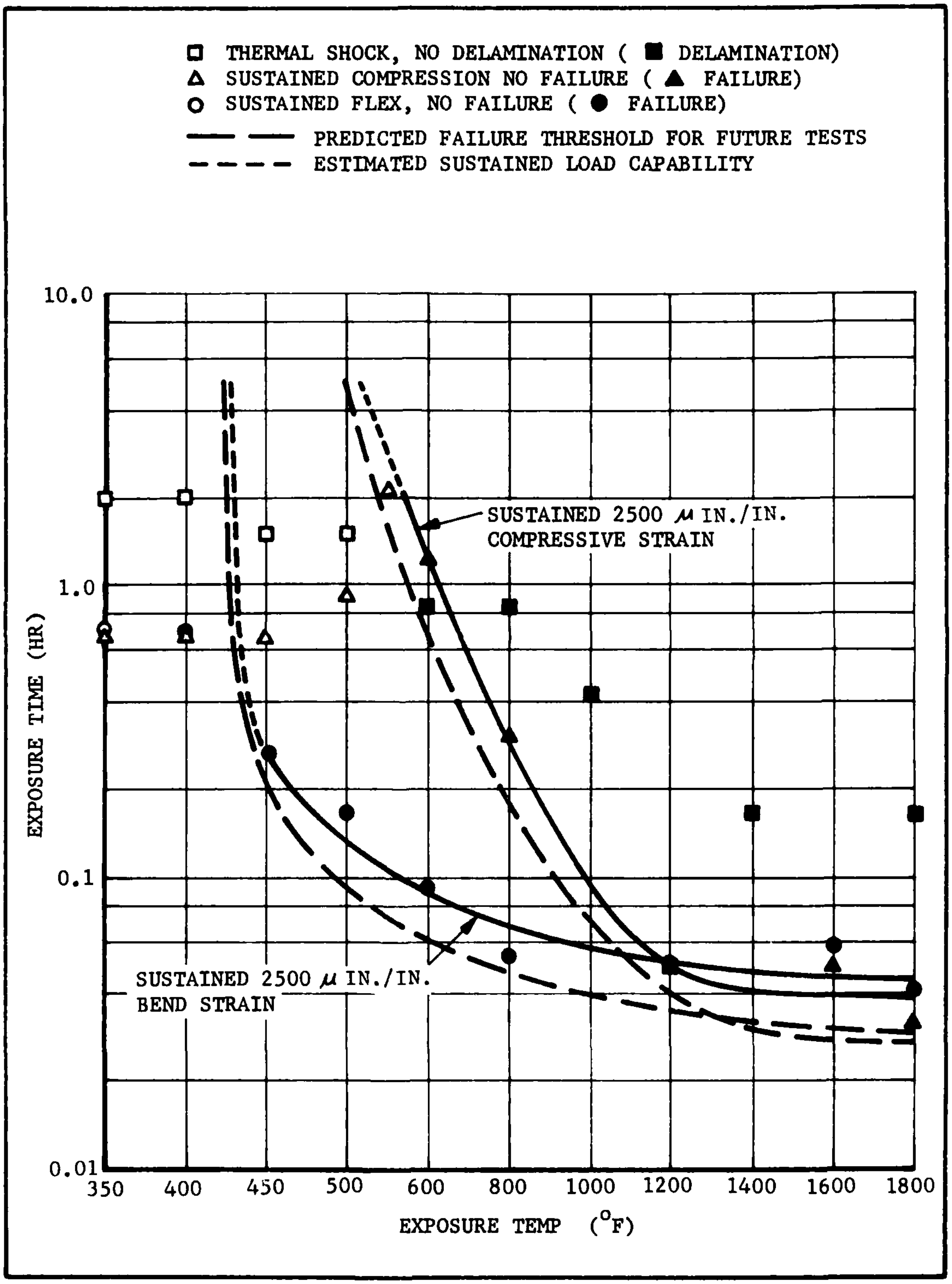


Figure 17. Comparisons of Sustained Load Tests During Thermal Shock

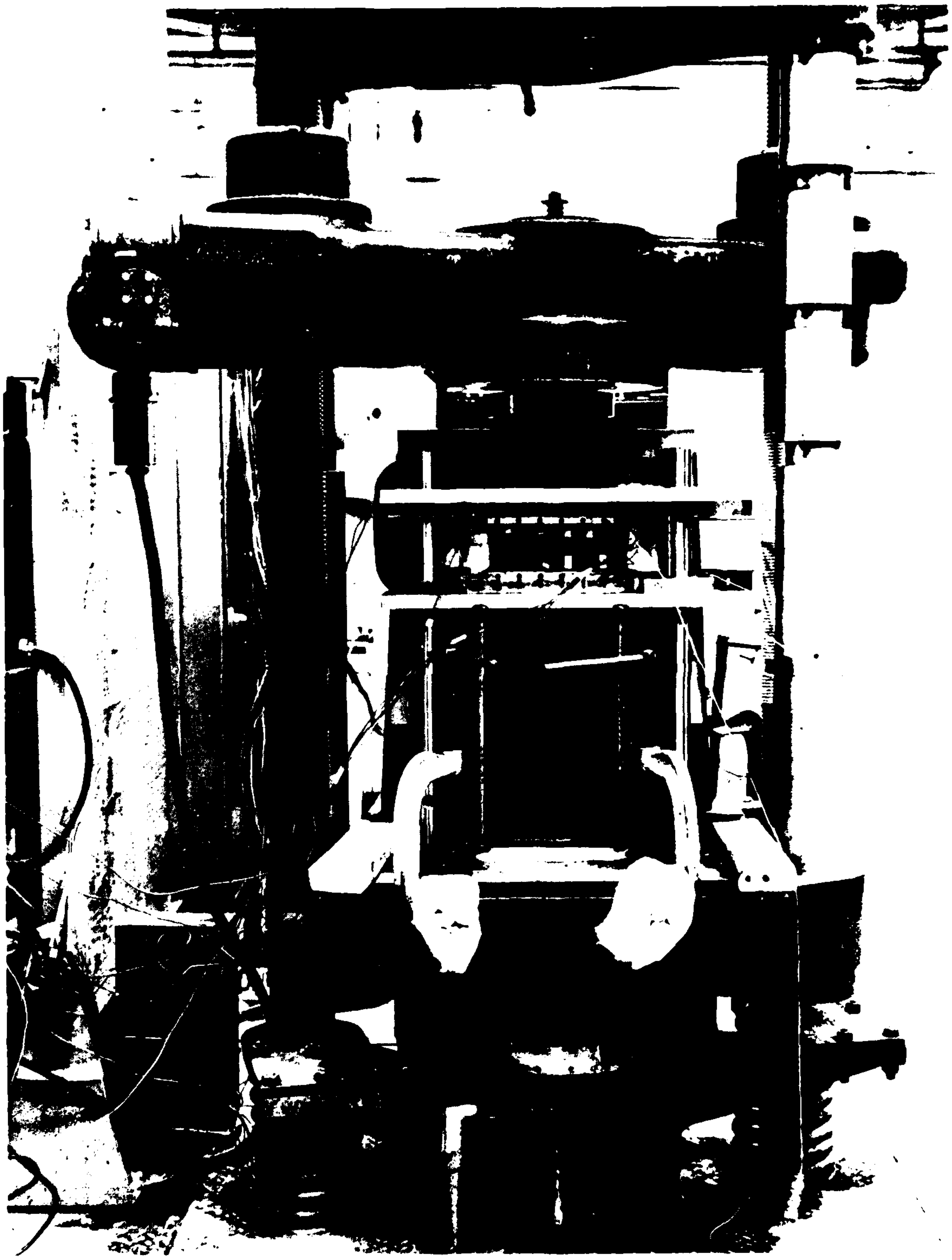


Figure 16. Sustained Compression/Thermal Shock Test Setup

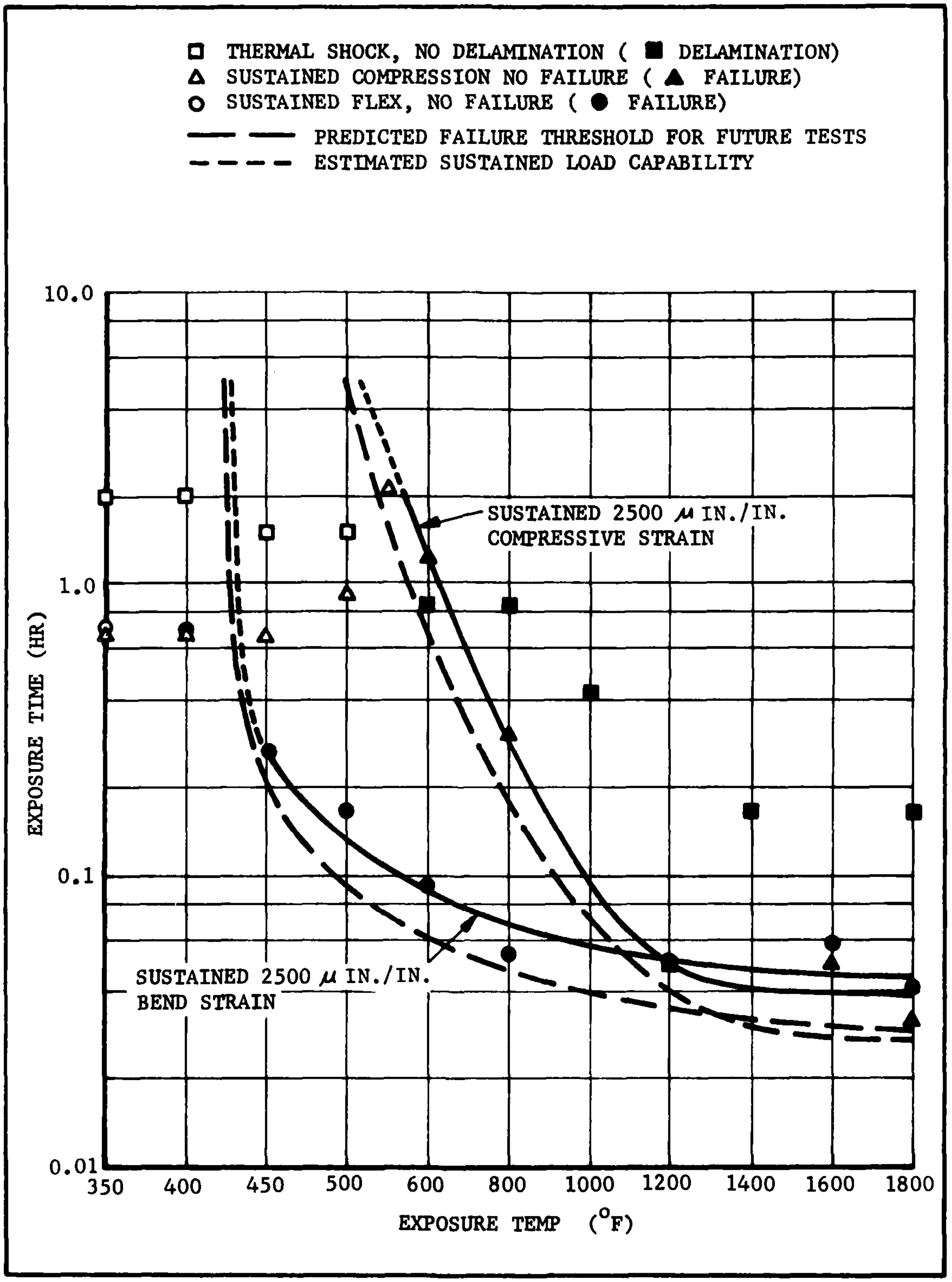


Figure 17. Comparisons of Sustained Load Tests During Thermal Shock

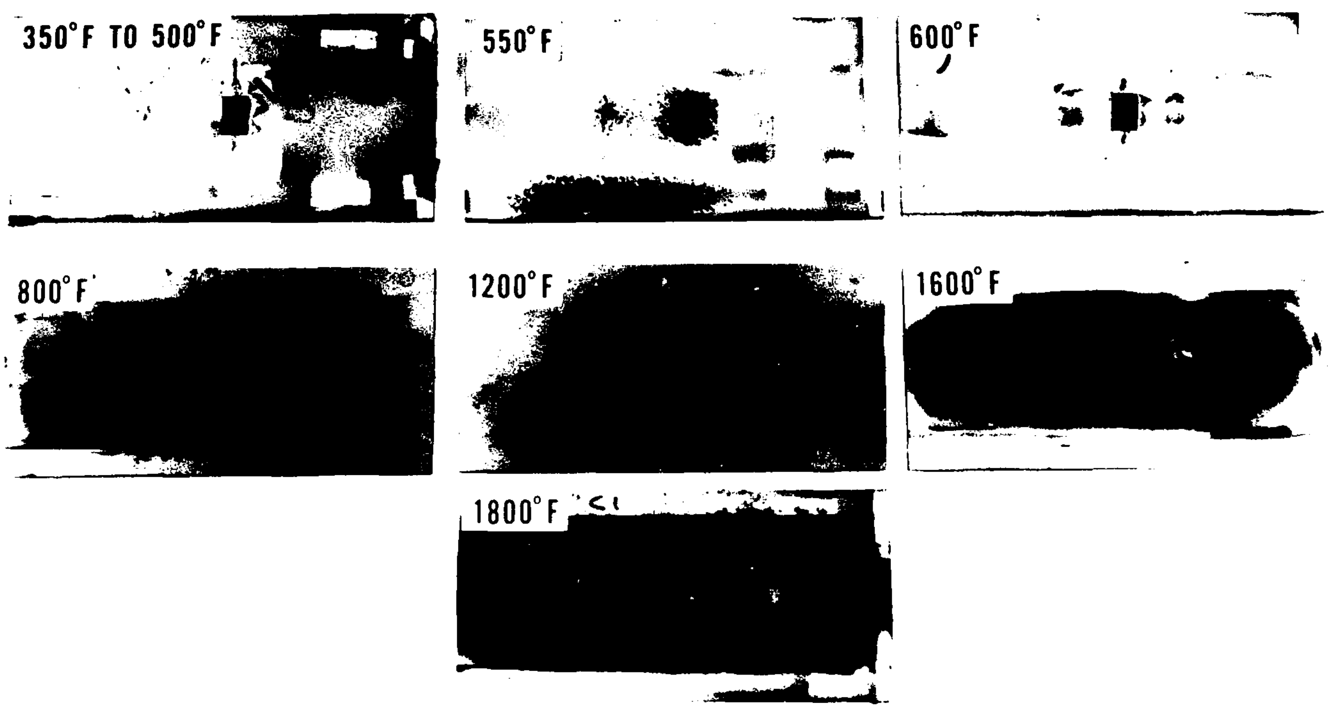


Figure 18. Sustained Compression Load Panels After Testing

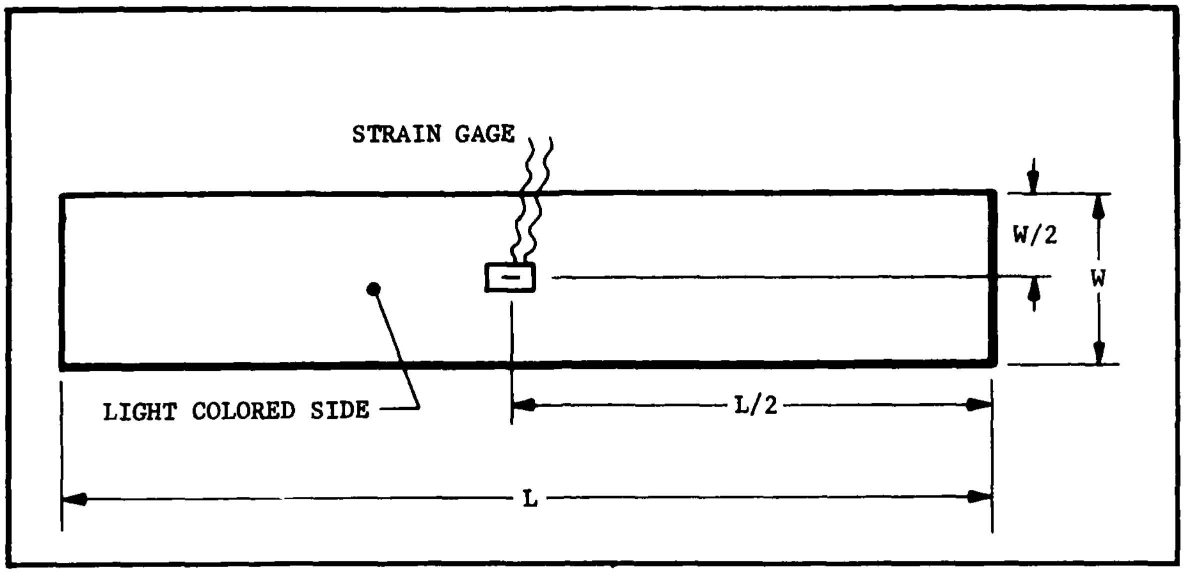


Figure 21. Strain Gage Location for Four-Point Flex Specimen

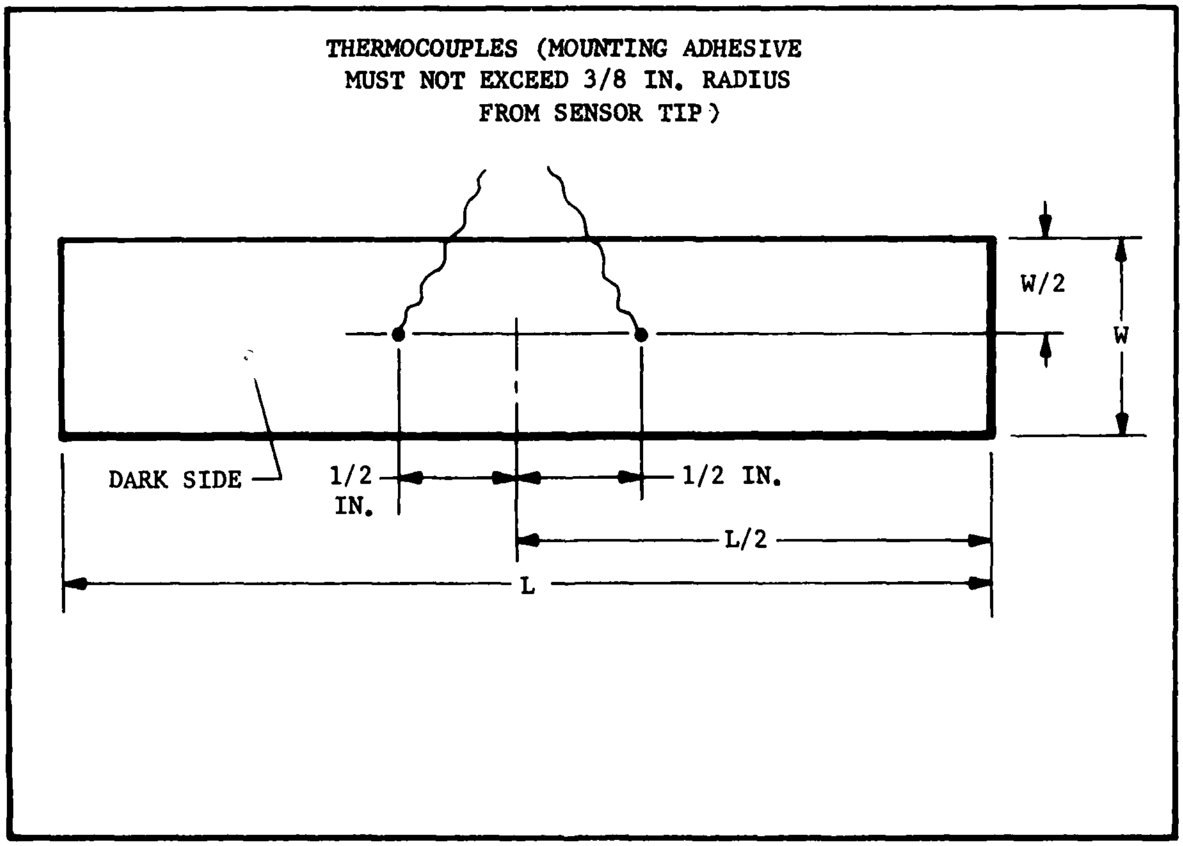


Figure 22. Thermocouple Location for Four-Point Flex Specimen

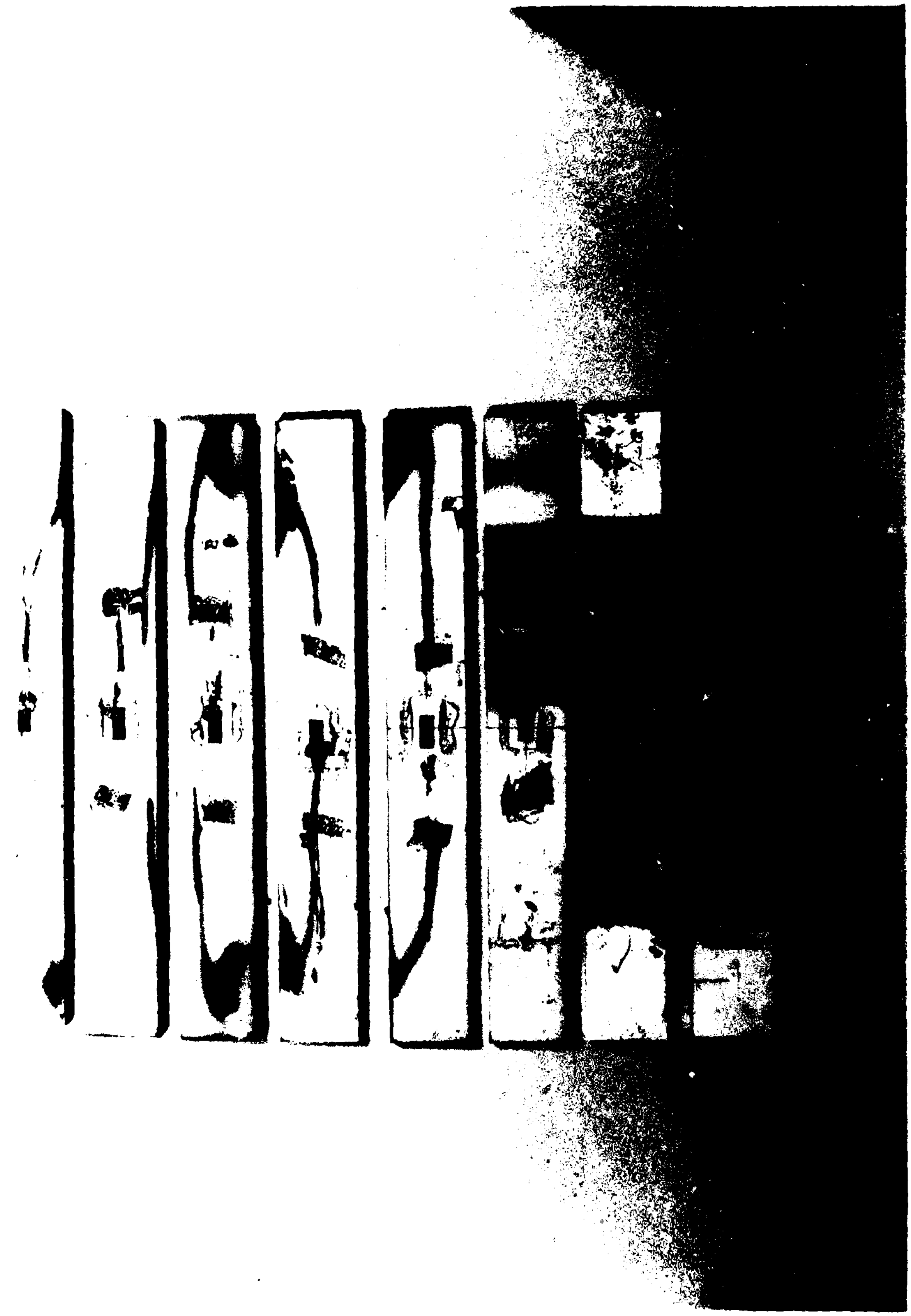


Figure 23. Sustained Four-Point Flex Specimens After Testing

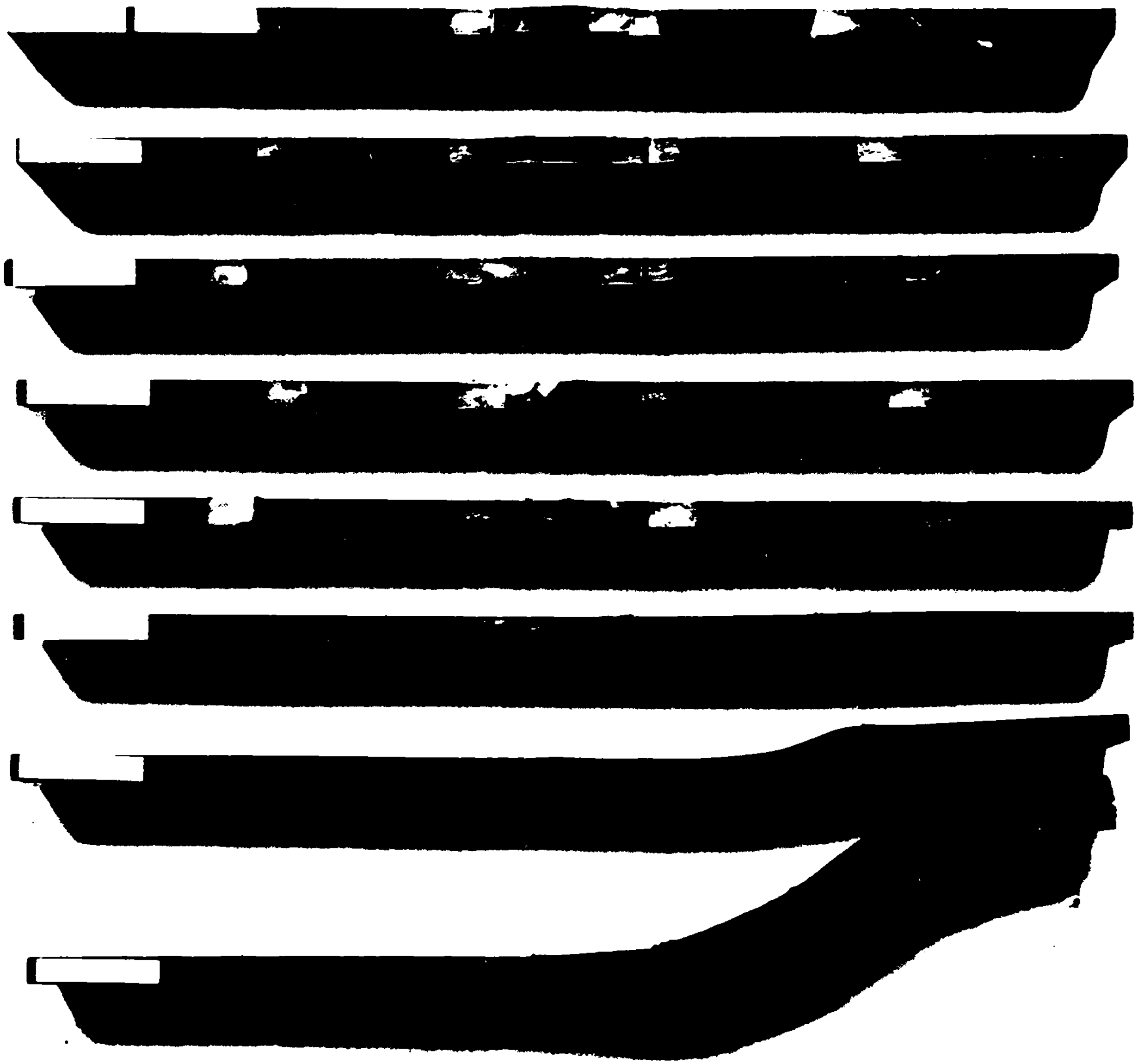


Figure 24. Edge View of Four-Point Flex Specimens After Testing

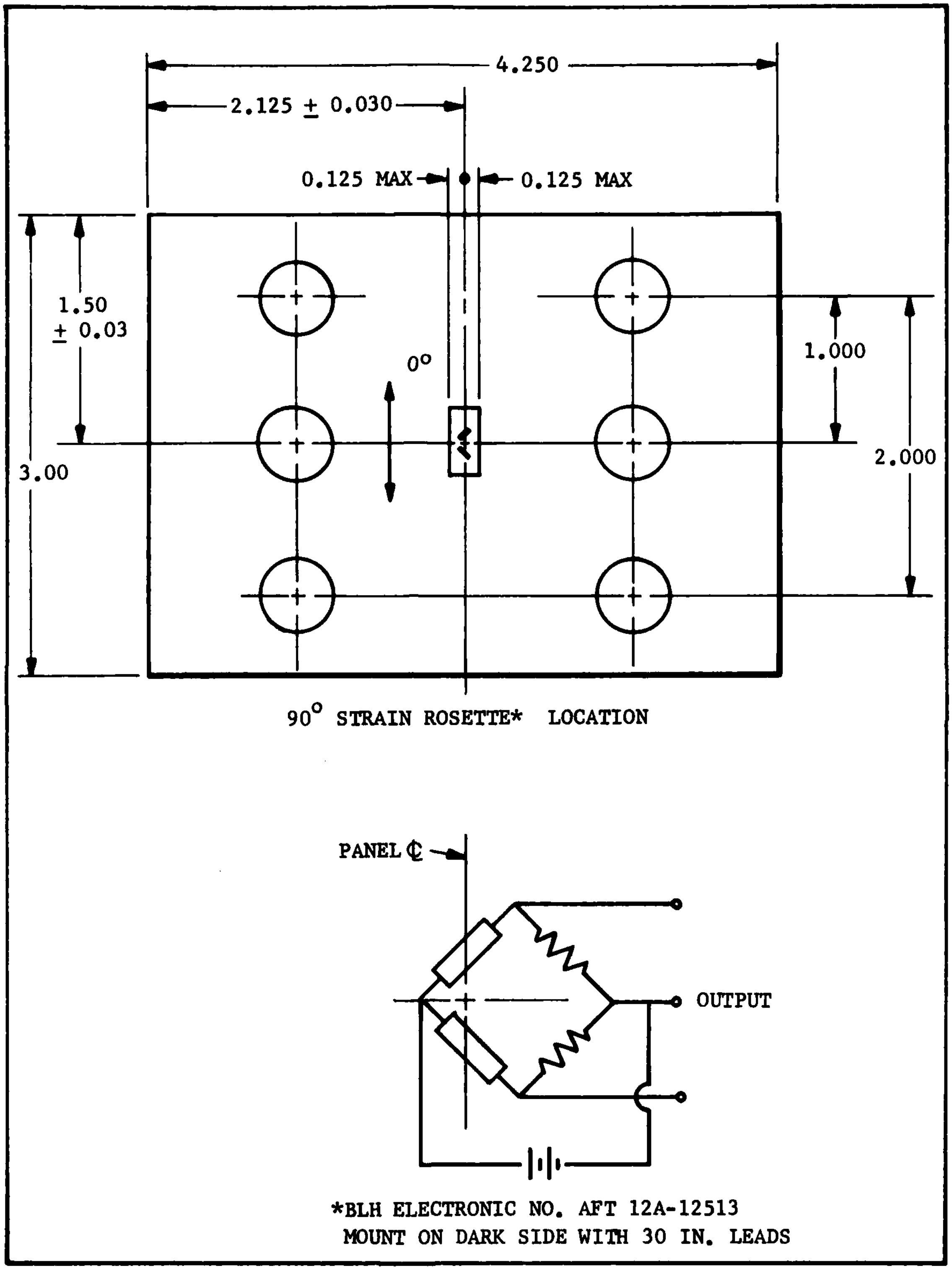


Figure 25. Original Rail Shear Specimen Configuration

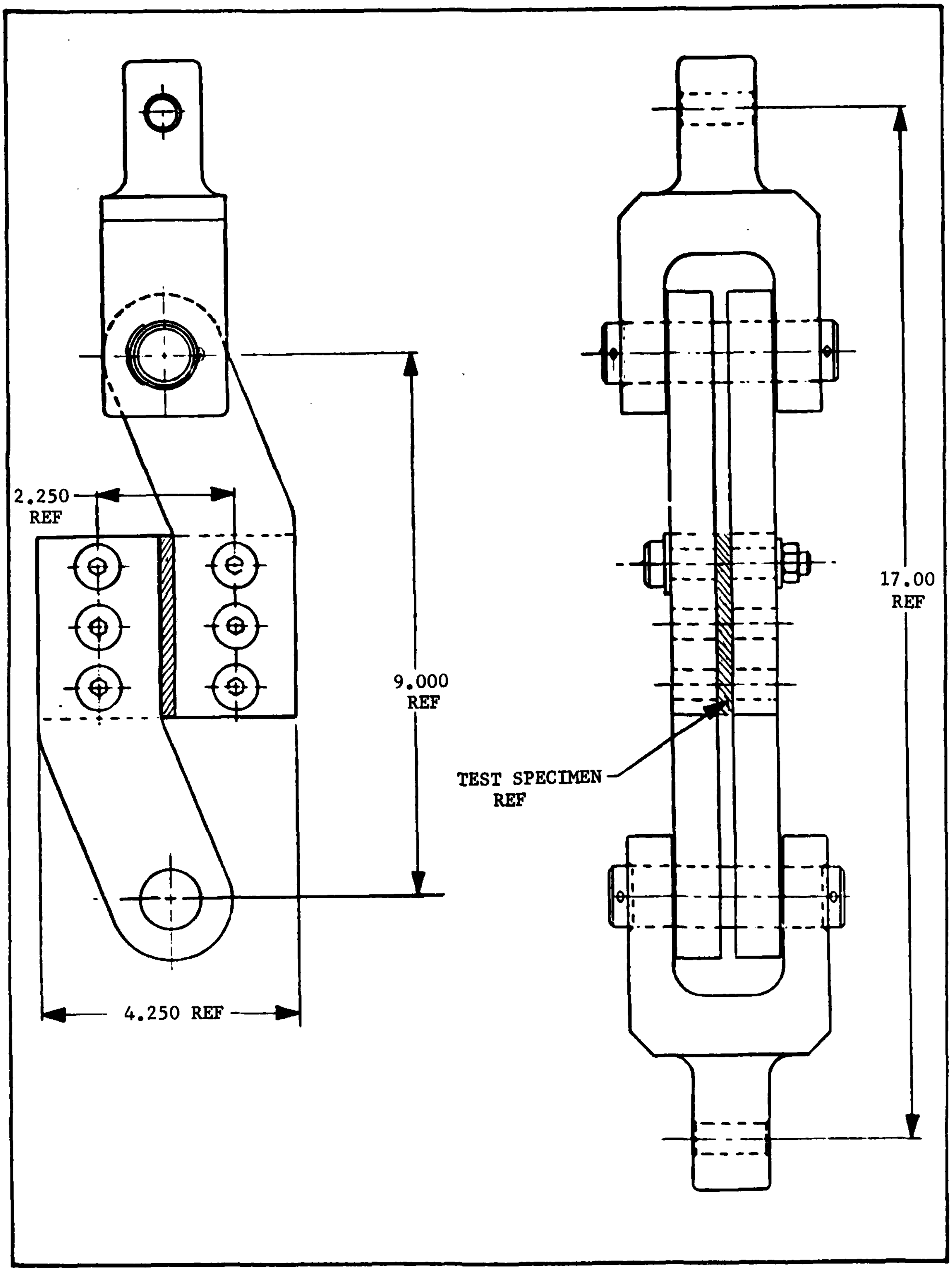


Figure 26. Rail Shear Fixture

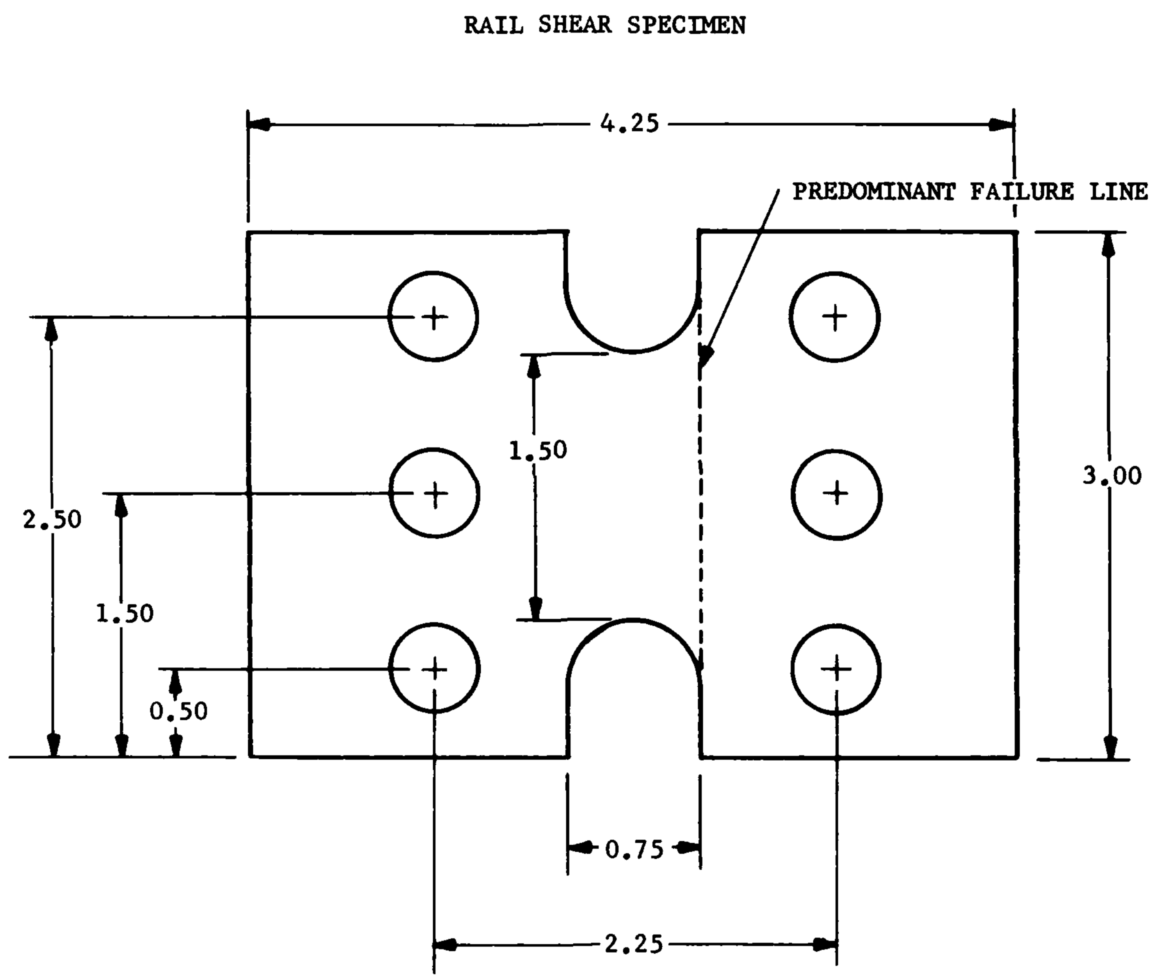


Figure 27. Modified Rail Shear Specimen

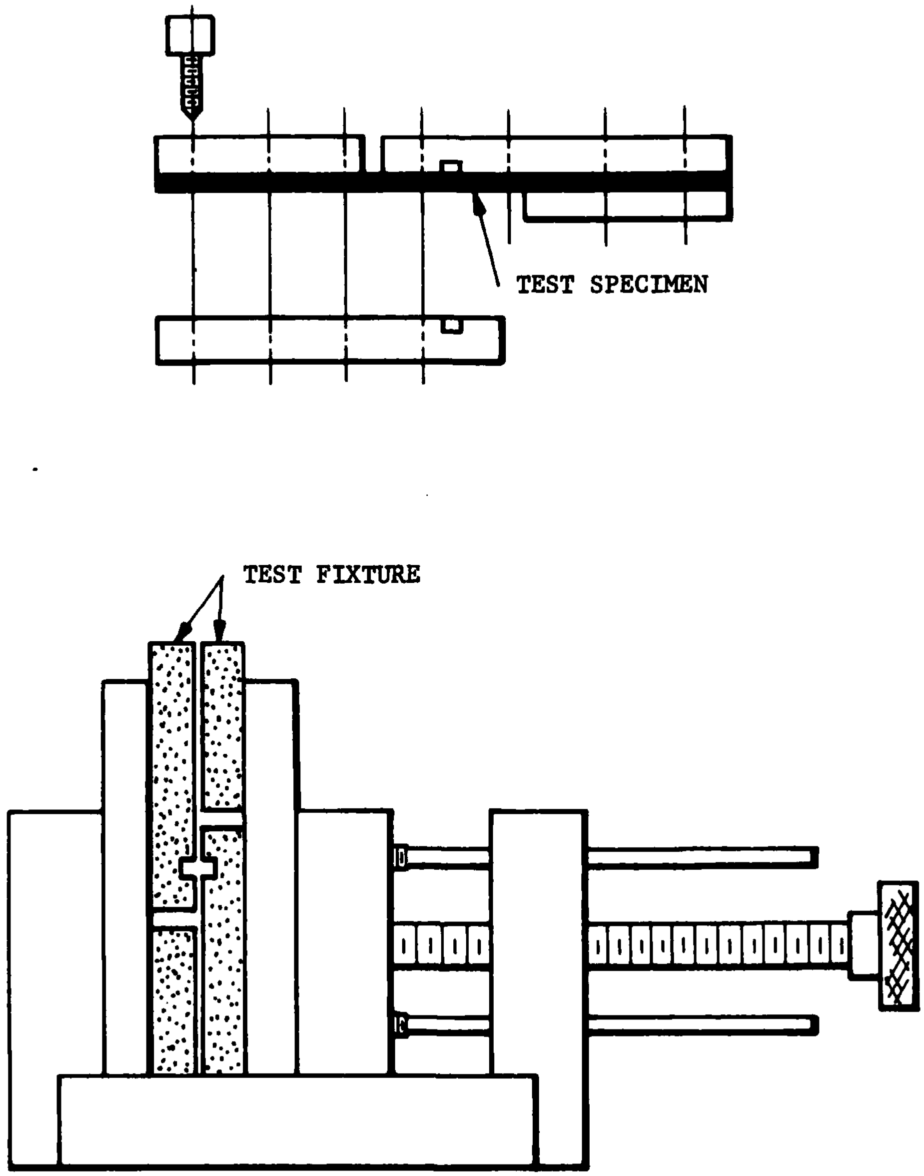


Figure 28. Compression Test Fixture Arrangement and Support Plates

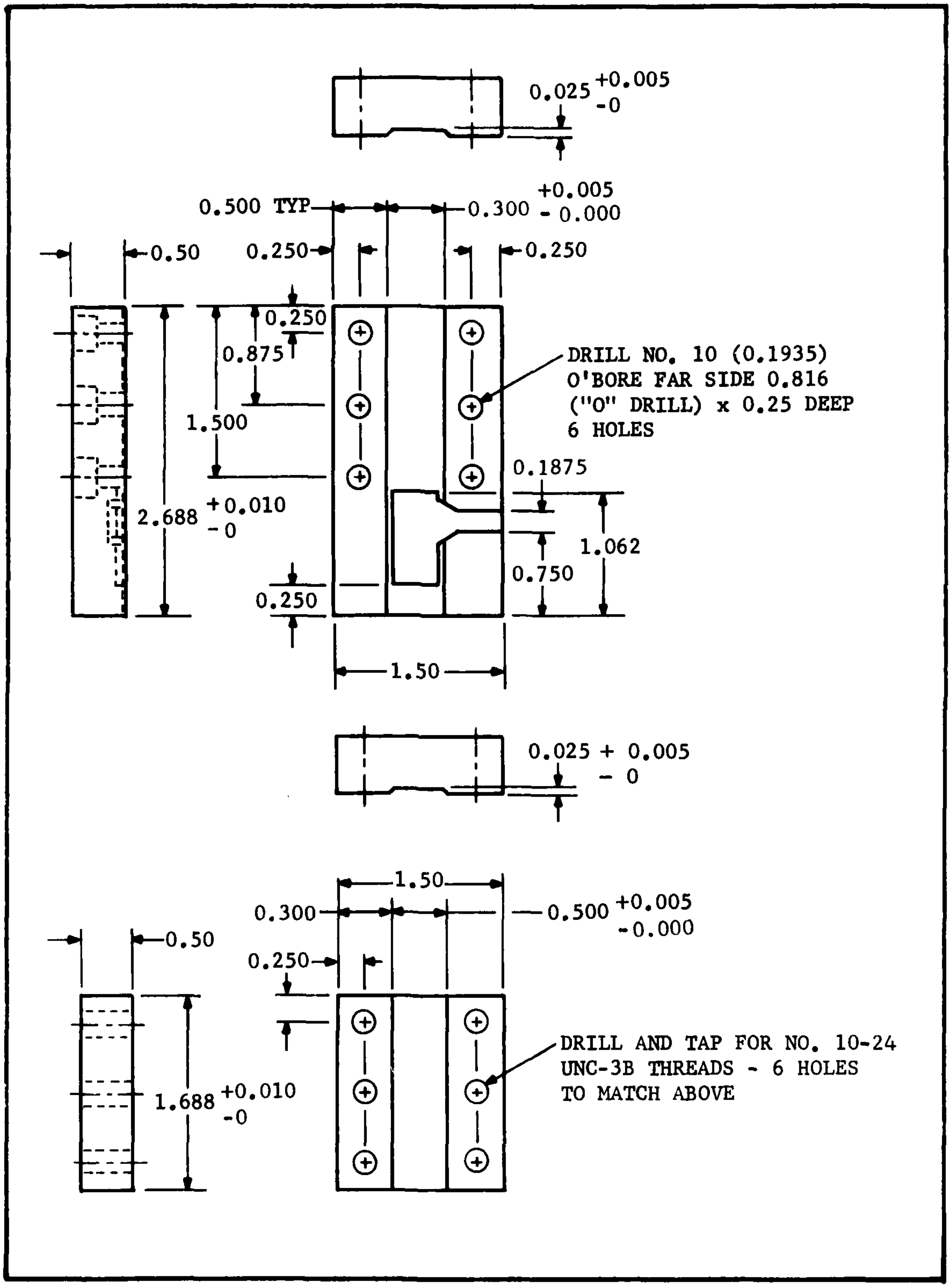


Figure 29. Support Plates for Compression Test Fixture

Figure 30. UTD Research Microscope

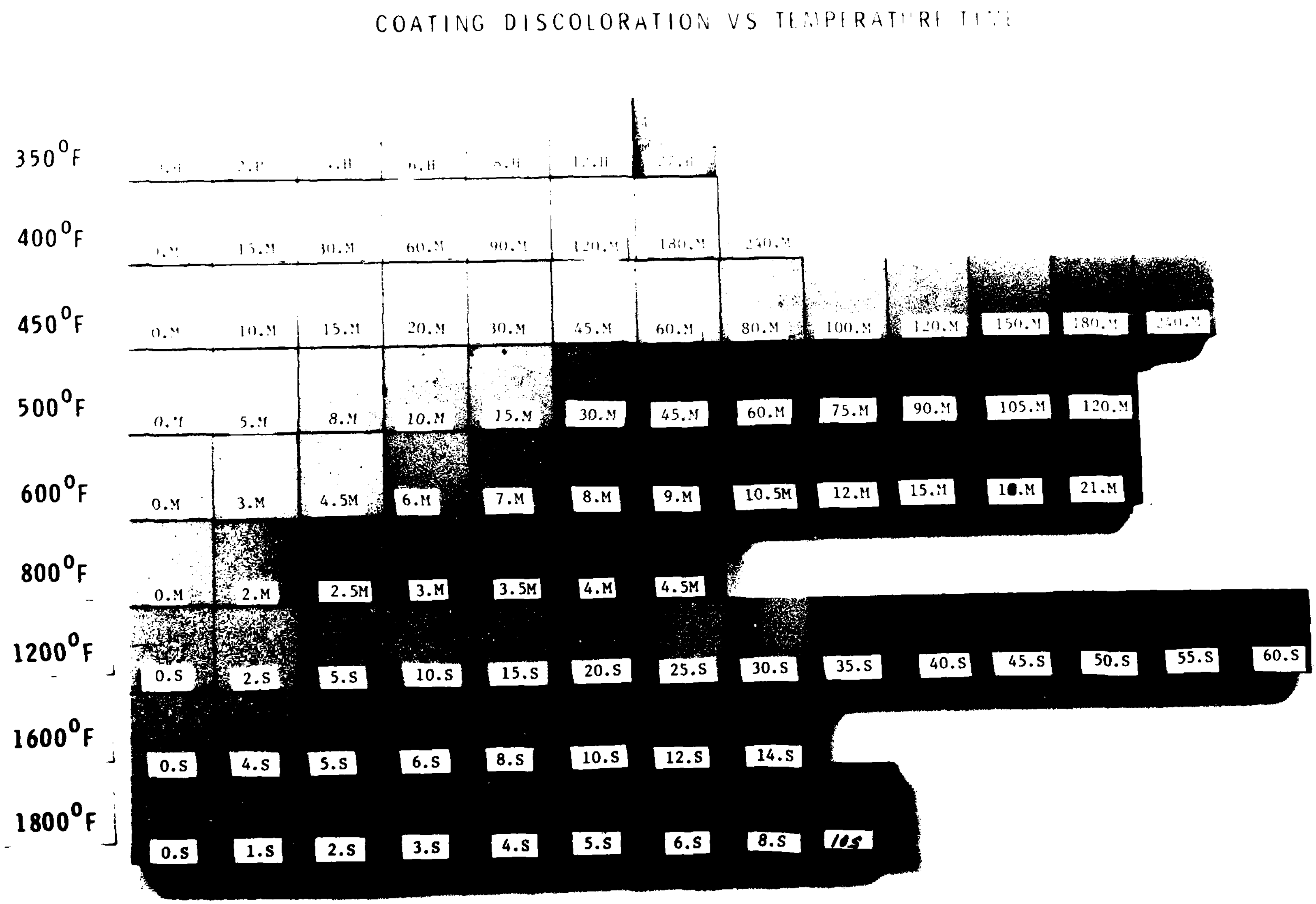


Figure 31. Discolorations of Top Coat Resulting From Thermal Exposure

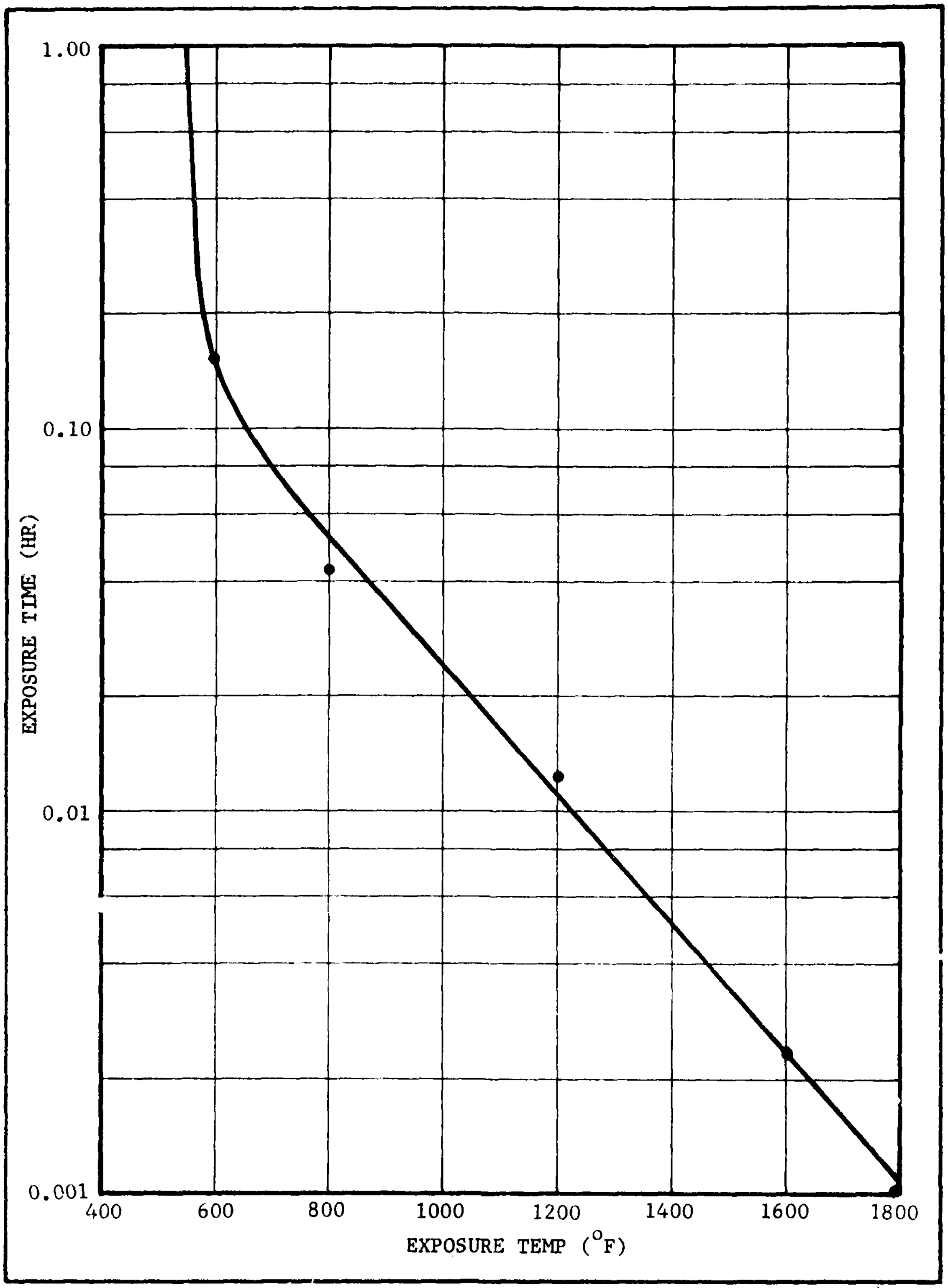


Figure 32. Predicted First-Ply Damage Based on Coating Color Changes for Exposure Time vs Temperature

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